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INVESTIGATIONS LEADING TO THE DEVELOPMENT  
OF A PRIMARY ZINC-SILVER OXIDE BATTERY  
OF IMPROVED PERFORMANCE CHARACTERISTICS

FINAL REPORT

Contract No. NAS 8-5493

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GEORGE C. MARSHALL SPACE FLIGHT CENTER  
Huntsville, Alabama

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XVIII, XX, XXI, XXII, XXIII

Abstract Card

Distribution List

## I. PURPOSE

The purpose of this contract was to generate design data making possible the construction of a reliable primary zinc-silver oxide battery of improved activated charge retention characteristics, greater voltage control, high energy density, increased temperature stability, and reduced gassing characteristics.

## II. ABSTRACT

Studies planned under four phases have been directed toward improving the primary zinc-silver oxide cell, with emphasis on close voltage regulation and extended retention-of-charge. Phases of study included (1) screening and evaluation of commercial separator materials, (2) preliminary cell design study, (3) evaluation of cell construction variables based upon a fractional factorial experiment and, (4) construction and evaluation of pre-prototype cells. The result has been an increased activated stand with no sacrifice in voltage or voltage regulation.

12500  
*A. L. H. H.*

### III. FACTUAL DATA AND DISCUSSION

#### A. Introduction

In accordance with the objective stated earlier, the following outline was set forth and served as a broad basis for studies relative to the primary zinc-silver oxide basis. A master schedule, contained in the Appendix, indicates the planned allotment of time for each phase of study and the progress attained.

#### Outline of Planned Investigations

##### 1. Evaluation of Separator Materials

###### a. Types

###### 1) Open

- a) Webril
- b) Viskon (R-35-D and R-75-D)
- c) Nylon (woven and non-woven)
- d) Polyethylene
- e) Polypropylene
- f) Polyvinyl Chloride
- g) Others

###### 2) Membranes

###### a) Cellulosic

- (1) 133 Visking
- (2) fibrous Visking
- (3) 300 Cellophane
- (4) 600 Cellophane

###### b) Inert

- (1) Permion
- (2) Acropor

###### 3) Special Treatments

- a) PVC dip
- b) PVA dip

###### b. Properties for Investigation

- 1) resistivity, ohm-cm<sup>2</sup>
- 2) speed of wetting
- 3) ability to retard silver migration
- 4) retention-of-charge characteristics as a function of temperature
- 5) absorbency

##### 2. Active Material Formulation

###### a. Positive Material

- 1) effect of apparent density
- 2) effect of formation current density on subsequent capacity efficiency
- 3) effect of electrolyte concentration on formation and capacity efficiency

- 4) treeing and migration of silver as affected by
      - a) temperature
      - b) electrolyte concentration
      - c) apparent density of divalent silver oxide
    - 5) efficiency of charge studies
    - 6) half-cell studies
  - b. Negative Materials
    - 1) efficiency of charge studies
    - 2) treeing and migration of zinc
      - a) as affected by temperature
      - b) as affected by KOH concentration
      - c) as affected by apparent density
    - 3) comparison of "spongy" and "metallic" zinc
    - 4) half-cell studies
- 3. Electrolyte
  - a. Effect of Concentration
    - 1) solubility of zinc
    - 2) solubility of divalent silver oxide
    - 3) electrolyte resistivity
  - b. Additives
    - 1) zinc oxide
    - 2) halide salts
    - 3) carboxymethyl cellulose
    - 4) starch
    - 5) lithium hydroxide
    - 6) lignin
    - 7) others
- 4. Cell and Battery Construction
  - a. Electrolyte Retention
    - 1) vent plugs
    - 2) head room
    - 3) separation
  - b. Voltage Regulation
  - c. High Rate Capabilities
  - d. Energy Density
  - e. Thermal Characteristics
- 5. Reaction Mechanisms
  - a. Gassing Rates
    - 1) effect of additives
    - 2) metallic as compared to spongy zinc
    - 3) treated spongy zinc
    - 4) electrolyte concentration
    - 5) electrolyte additives
    - 6) effect of grid material
  - b. Theoretical Investigation
    - 1) reaction mechanisms of gassing
      - a) gassing at positives
      - b) gassing at negatives
    - 2) establish theoretical hypothesis for preventing gassing



## 6. Reliability Evaluation

- a. Failure Effects Analysis
- b. Drift Analysis on Parts
- c. Stress Analysis and Reliability Estimation for Each Part.

## B. Goals of Contract NAS 8-5493

Certain major goals were set forth in accordance with Contract No. NAS 8-5493. These are listed below:

- 1. Plateau voltage of 1.40 volts per cell when discharged at the twenty (20) minute rate;
- 2. Maximum possible capacity;
- 3. Good voltage control;
- 4. High energy density;
- 5. Minimum of thirty days activated stand at room temperature;
- 6. Low gassing rates as required for space applications.

## C. Phase I - Separator Evaluation

### 1. Objectives

The ideal separator would have the following characteristics:

- a. low electrical resistance
- b. high absorbency
- c. stability in concentrated potassium hydroxide
- d. resistance to oxidation (caused by charged positive plate)
- e. high oxygen permeability (less important in the primary cells)
- f. ability to retard migration of silver and zinc ions
- g. good physical strength
- h. rapid wetting ability
- i. stability of all properties over wide temperatures.

### 2. Physical Testing

#### a. Materials Tested

Table No. I lists separators, both membranes and "open" types which were tested, as well as their identifying features.

TABLE NO. I

SUMMARY OF COMMON SEPARATORS

MEMBRANES	WET THICKNESS (inches)	DESCRIPTION
133 Visking	0.009	Cellulosic
Fibrous Visking	0.009	Fibrous sausage casing
300 Cellophane	0.003	Regenerated cellulose
600 Cellophane	0.0042	Regenerated cellulose
PE Film 10/20	0.002	Irradiated polyethylene
PVA Film	0.002	Poly (vinyl alcohol)
OPEN TYPES	WET THICKNESS (inches)	DESCRIPTION
Nylon	0.003	Woven 100% Nylon
Nylon	0.005	Woven 100% Nylon
Nylon	0.011	Woven 100% Nylon
Pellon	0.005	Non-woven Nylon
Pellon	0.011	Non-woven Nylon
R-35-D	0.00575	Non-woven Nylon
R-75-D	0.0105	Non-woven Nylon

b. Testing Procedures(1) Electrical Resistance

The apparatus used for determining areal resistance consists of a plastic reservoir filled with electrolyte and having parallel inert metallic electrodes at either of two opposing sides. This reservoir accepts a sample-holder which allows current to flow through an opening of 8.98 square inches. To determine resistance, the separator being tested is inserted and secured into the sample-holder, and direct current is passed through the apparatus at a measured rate. The voltage drop across the apparatus is measured. This measurement is repeated as a blank, i.e., with no separator across the sample-holder opening. In this manner, effects of minor temperature variations may be eliminated. From the difference in the above voltage drops ( $\Delta E$ ), the areal resistance ( $R$ ) can be calculated according to the formula

$$R = \frac{(\Delta E)(8.98 \text{ in}^2)}{25 \text{ amperes}}, \text{ where the current through the apparatus is}$$
 twenty-five (25) amperes. The resistance is, of course, in units of ohm-inch<sup>2</sup>.

Data have been accumulated relative to electrical resistance of single and multiple layers of both "open" and membranous separators. These data are revealed by Table No. II.

TABLE NO. II

RESISTANCE OF MEMBRANES  
(Multiple layers at +80° F)

SEPARATOR	RESISTANCE (ohm-inch <sup>2</sup> )		
	TWO LAYERS	THREE LAYERS	ONE LAYER
PE 40/20	.02255	.0628	.0056
300 Cellophane	.01796	.0257	.0054
.0015" PVA	.0287		.0072
600 Cellophane	.01796	.323	.0117
133 Visking	.0898	.531	.0226
Fibrous Visking			.0154
Polypor			.0826
.004" PVA			.0180
	<u>Absorbents</u>		
R-35-D Viskon	.0055		.0084
Pellon	.0617		.0116
.004" Polypropylene			.0107
.009" Polypropylene	4.22		.0226
R-75-D Viskon	.0226		.0112
9526 Nylon	.0898		.00557
Webril	.0561		.0055
Dynel			.1123

Two significant facts have become apparent during resistance studies: (a) Total electrical resistance of multiple layers of membranes does not increase linearly with increasing numbers of layers. For instance, one layer of 300 Cellophane exhibits a resistance of 0.0053 ohm-inch<sup>2</sup>, while two and three layers display resistances of 0.0180 and 0.0257 ohm-inch<sup>2</sup>, respectively; (b) Certain "open" separators exhibit a higher resistance than might be expected. R-35-D, a non-woven absorbent, exhibits a resistance greater than that of a single layer of either PE film, 300 Cellophane, or PVA film, although it has a much greater gas permeability. It is likely that gases produced at the inert electrodes adhere to the "open" separator samples, reducing the available current path and causing relatively higher resistances. It should be noted, however, that primary cells are not ordinarily electrolyte-flooded, thus the effects of gas entrapment do not invalidate the test data.

(2) Cycle Testing of Separation

Certain combinations of separator materials have also been evaluated by life cycling positive-limited three-plate test cells. Cycles consisted of 61.5 minutes charge at 0.76 ampere constant current, and 8.5 minutes discharge at a 5-ampere rate. This corresponds to a discharge depth of slightly less than 20% of the theoretical divalent silver capacity of the cell, with a 10% overcharge on each cycle. The use of cadmium rather than zinc negative material eliminates the likelihood of cycle failure because of loss of active material. This insures

that the number of cycles to failure of an individual cell indicates the relative ability of the particular separator combination to withstand the deleterious environmental conditions encountered in the alkaline battery system.

While the number of cycles to failure is of no specific value in itself, comparison of cycle life of various test cells reveals the relative quality of individual separator combinations and aids in selecting the optimum for use in the primary cell. This test serves to accelerate evaluation of separator materials under actual conditions of service whereby the most promising types may be selected for studies in conventional cell assemblies.

Table No. III lists the combinations of separators which were tested, as well as displaying the results. Periodic end-of-charge and end-of-discharge voltages were recorded. At fixed intervals, the test cells were allowed to remain in the charged state for 48 hours for the purpose of monitoring open circuit voltages. The criterion for failure was reversal upon discharge or an open-circuit voltage lower than that associated with the cadmium-monovalent silver oxide couple following the stand period.

### (3) Speed of Wetting

In the procedure used for determination of separator resistance, readings are taken at time intervals until no further change in resistance is noted. This allows observation of the speed of wetting of the various materials. As there is no widely used criterion for description of this property, data have been interpolated to reveal the soak time ( $TR_2$ ) after which a separator displays a resistance equal to twice its final stable value. These wetting speeds are, of course, only realized under ideal conditions of electrolyte accessibility. Wetting speed in a cell can vary greatly with cell design. Table No. IV reveals data relative to speed of wetting as well as the effect of temperature on this quality.

### (4) Electrolyte Absorption and Resistance

Another important quality of separators is electrolyte retention. To evaluate this characteristic, samples of various separators were dried, weighed accurately, soaked for 72 hours in 1.300 specific gravity potassium hydroxide solution, removed, and reweighed. Before the final weighing, the sample was held vertically a few seconds until the excess KOH formed droplets at the edge of the sample. Only these droplets were blotted to avoid removing electrolyte held in the pores of the sample. A summary of absorption data is presented in Table No. V.

Another importance of electrolyte absorption lies in the determination of the optimum quantity of electrolyte to be included in a cell. It is anticipated that the optimum quantity of electrolyte for this system is that which saturates both the plates and separator. However, cell tightness will affect the absorption properties of separator materials.

### c. Conclusions Relative to Separator Evaluations

1. A number of separator materials are capable of performing satisfactorily for a period of months at room temperatures. The exact life, of course, of a separator system is a function of the number of layers between plates.
2. Low electrical resistance of the cellulosic membranes offers a distinct advantage over PE films or Visking on a layer-for-layer basis. Absorbency of cellophane is also greater than that of either PE films or Visking.
3. PE film is not as effective in preventing silver migration as cellophane, although as a modified polyethylene, it is essentially inert to the caustic environment.

### D. Phase II - Preliminary Testing and Control Cell Evaluation

#### 1. Objective

The purpose of this phase of study was to select the most promising separator combination from four feasible combinations. This combination would then be held constant in the evaluation of cell construction variables itemized in the Outline of Planned Investigations (III, A.1). It was also anticipated that this phase of study would indicate the order of magnitude of activated life for the medium-rate primary zinc-silver oxide cell at room temperature.

#### 2. Procedure

##### a. Separator Combinations Tested

Five cells were constructed with each of four separator combinations as listed below:

##### 1) Group I, Cells 1-1 to 1-5

One wrap Polypor next to the positive.  
Two wraps 300 Cellophane over the Polypor.  
One wrap R-35-D absorbent over the negative.

##### 2) Group II, Cells 2-1 to 2-5

One wrap #9526 Nylon next to the positive.  
Three wraps PE film over the Nylon.  
One wrap R-35-D on the negative.

##### 3) Group III, Cells 3-1 to 3-5

One wrap #9526 Nylon next to the positive.  
Two wraps 300 Cellophane over the Nylon.  
One wrap R-35-D on the negative.

##### 4) One wrap 133 Visking on the positive. One wrap R-75-D on the negative.

These combinations were employed because they represent approximately equivalent total thicknesses of separator materials in each cell.

#### b. Cell Testing

All cells were activated with 20 cc of 1.400 specific gravity potassium hydroxide solution. One cell of each type was discharged at 37 amperes following a 2-hour soak period (this rate is equivalent to 0.815 ampere per square inch of positive surface area, or the 15 to 20-minute rate). Remaining cells were discharged following activated stand periods of two weeks, three weeks, one month and two months. Results of these discharges are illustrated by Figure Nos. 1 through 4.

#### 3. Conclusions

All cells displayed a significant decrease in capacity after one month stand, although they performed satisfactorily. Only Series IV cells displayed a continued decrease in capacity in the interval between one and two months activated stand. Data relative to capacity as a function of activated stand are displayed by Figure No. 5. Smooth curves were not employed in representing these data because present knowledge is not sufficient to formulate an explicit function relating these variables.

Three layers of the PE film (an ion exchange membrane) proved to be undesirable for this specific application. Figure No. 2 reveals cells employing three layers of PE film in addition to 9526 Nylon on the positive displayed in general low voltage and poor voltage control. Post-mortem of PE cells revealed that all layers of the film contained silver. Two layers apparently would not have been sufficient to prevent silver from reaching the zinc plate, indicating that decreasing the number of wraps of this membrane to reduce cell resistance would not be an acceptable approach to improving voltage characteristics.

Use of one layer of Polypor, another ion-exchange membrane, along with two layers of 300 Cellophane, revealed no outstanding advantages. The Polypor membrane did not appreciably slow silver migration. The cellulosic membrane showed considerable silver loading and physical deterioration.

The combination of 9526 Nylon with two wraps of 300 Cellophane appeared to be the most satisfactory one. Both voltage and capacity were very good at the two and three week stand levels. Voltage and capacity for the greater stand periods were comparable to that yielded by other separator combinations.

With the exception of the discharge following two months stand, least variation of voltage and capacity with time was exhibited by the Control, or Series IV cells. This cell employed one layer of 133 Visking next to the positive plate. Voltage regulation was not favorable, however.

Results of cell examination verified the advantages of employing an "open" material next to the positive plate. This prevents damage to the membrane by crystalline formations. These formations otherwise tend to force the

separator from the plate, causing uneven discharge of the positive plates and tearing or puncturing the membrane.

The tests conducted under Phase II indicated that under carefully controlled conditions, individual cells might stand satisfactorily for a period of weeks, although a precise estimation of reliability cannot be made. Even greater doubt would be cast on any estimation of battery reliability because of thermal characteristics and other variables.

#### E. Phase III - Fractional Factorial Study

##### 1. Objective

The Phases I and II having indicated a satisfactory combination of separators and method of separation, it was decided to proceed with evaluation of other variables listed in the Outline of Planned Investigations.

The objective of Phase III was to evaluate the effects of combining levels of several variables in full scale test cells.

##### 2. Procedure

###### a. Variables

Table No. VI lists test variables and levels which were selected for study.

TABLE NO. VI

TEST VARIABLES AND THEIR LEVELS

A. Additives to Electrolyte

A<sub>0</sub> - None  
A<sub>1</sub> - 1% gel  
A<sub>2</sub> - MnO<sub>2</sub> (at saturation)  
A<sub>3</sub> - LiOH (at saturation)

B. Electrolyte Concentration

B<sub>0</sub> - 35%  
B<sub>1</sub> - 40%  
B<sub>2</sub> - 45%

C. Positive Material Density (gms./cu.in.)

C<sub>0</sub> - 68  
C<sub>1</sub> - 74  
C<sub>2</sub> - 80

D. Positive Grid Metal

D<sub>0</sub> - 4/0 Ni  
D<sub>1</sub> - 4/0 Ag

E. Negative Material Density

E<sub>0</sub> - 40  
E<sub>1</sub> - 45  
E<sub>2</sub> - 50

F. Additive Content in Negative Plate \*

F<sub>0</sub> - 1%  
F<sub>1</sub> - 2%  
F<sub>2</sub> - 4%

G. Negative Grid Metal

G<sub>0</sub> - Copper (4/0)  
G<sub>1</sub> - Silver Flashed Cu (4/0)  
G<sub>2</sub> - Silver (4/0)

H. Negative Formulation \*\*

H<sub>0</sub> - Pasted \*\*\*  
H<sub>1</sub> - Sponge  
H<sub>2</sub> - Metallic

\* The nature of this additive and its introduction into the negative plate is proprietary.

\*\* These are descriptive names referring to three types of negative plates prepared by proprietary procedures.

\*\*\* It proved impractical to electroform pasted negative material in sufficient quantity and of the quality required by this experiment.



## b. Anticipated Effect of Variables

### 1) Additives to the Electrolyte (Factor A)

Effects of lithium hydroxide have been reported in considerable depth under other contracts, but primarily relative to the secondary system. Its effect would be expected to be increased charge retention.

Manganese dioxide, sparingly soluble in caustic solutions, might retard hydrogen liberation at the negative plate.

The gelatinous electrolyte additive was expected to retard bulk movement of electrolyte and discourage relocation of zinc throughout the cell.

### 2) Electrolyte Concentration (Factor B)

Characteristics of the KOH-H<sub>2</sub>O system as a function of "strength" and temperature are exhibited by Figure Nos. 6 and 7. It is common knowledge that resistivity of a KOH-H<sub>2</sub>O solution is at a minimum in the region of 1.3 specific gravity, or 31% by weight of KOH in water. A cell employing a 31% solution of KOH as electrolyte would be expected to display the highest voltage at any temperature or current density of discharge. The freezing point of the electrolyte is also at a minimum for this strength as is indicated by the phase diagram, Figure No. 6.

Solubility of the silver species is also greatest at this concentration; therefore, higher concentrations are commonly employed in cells intended for long shelf life or high temperature stands.

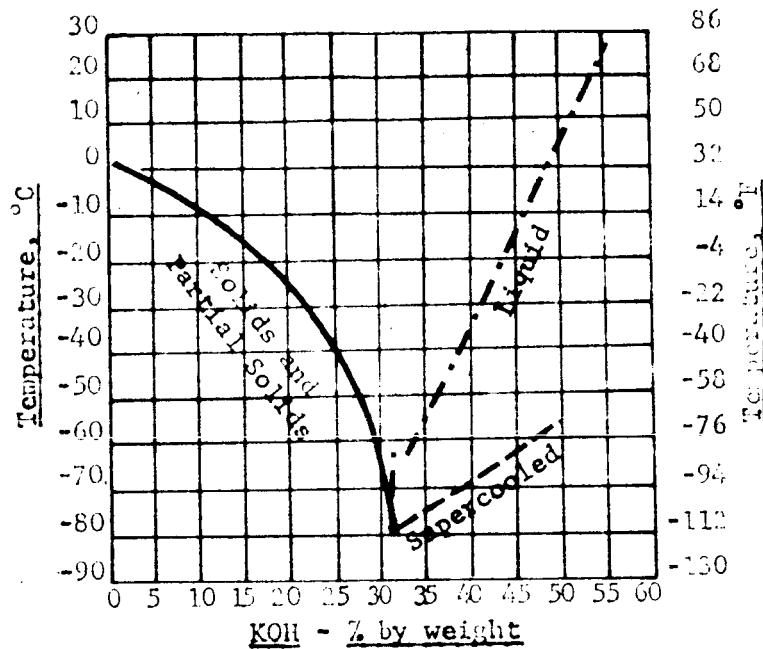


FIGURE NO. 6  
KOH-H<sub>2</sub>O PHASE DIAGRAM

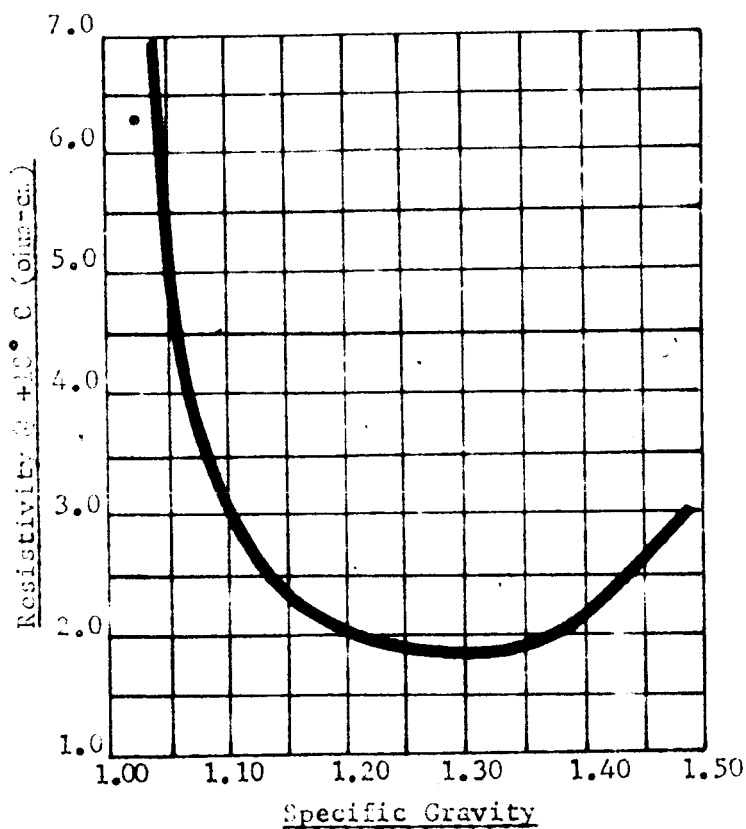


FIGURE NO. 7  
RESISTIVITY OF  
KOH-H<sub>2</sub>O SOLUTION

### 3) Positive Material Density (Factor C)

Thickness to which the positive plate is compressed affects the mechanical bonding between particles as well as the effective surface area of the resulting plate. These changes are reflected in the availability of the potential electrochemical energy, and hence, the capacity efficiency of the positive active material.

### 4) Positive Grid Material (Factor D)

Choice of grid metal determines the electrical resistance of the grid. This is reflected in the discharge voltage and possibly, capacity retention.

The following is a discussion of the relative current-carrying capacities of various commonly used grid materials. An elementary formula for the resistance of a conductor is

$$R = P \frac{L}{A} \quad \text{where}$$

P, L, and A are the resistivity, length, and area, respectively, of the conducting material. We shall assume that a current flows unidirectionally in the grid, in the direction of the terminal. Taking a unit length of one inch in the direction of current travel, the resistance may easily be determined for a square inch of grid metal. Table No. VII contains data relative to these determinations.

TABLE NO. VII

#### RESISTANCES OF COMMON METALLIC GRIDS

GRID METAL	RESISTIVITY	IN <sup>3</sup> METAL PER SQ. IN. OF GRID	OHMIC RESISTANCE OF 1 IN <sup>2</sup>	RESISTANCE RELATIVE TO 4/0 Ag R'
4/0 Ag	1.62 x 10 <sup>-6</sup> ohm-cm	0.00244	1.69 x 10 <sup>-3</sup>	1.00
3/0 Ag	1.62 x 10 <sup>-6</sup> ohm-cm	0.00192	2.14 x 10 <sup>-3</sup>	1.27
2/0 Ag	1.62 x 10 <sup>-6</sup> ohm-cm	0.00157	2.62 x 10 <sup>-3</sup>	1.55
1/0 Ag	1.62 x 10 <sup>-6</sup> ohm-cm	0.00069	5.96 x 10 <sup>-3</sup>	3.53
4/0 Cu	1.72 x 10 <sup>-6</sup> ohm-cm	0.00295	1.48 x 10 <sup>-3</sup>	0.876
4/0 Ni	7.24 x 10 <sup>-6</sup> ohm-cm	0.00171	1.03 x 10 <sup>-2</sup>	6.38
4/0 Zn	5.92 x 10 <sup>-6</sup> ohm-cm	0.00371	4.06 x 10 <sup>-3</sup>	2.40

These data are not absolutely correct because of the cross-sectional area of the component wire variance with the type grid. It is readily apparent, however, that 4/0 nickel grid displaces more active material than 2/0 silver, but has a resistance more than 2/0 silver grid and 6.38 times that of 4/0 silver. Although copper is slightly more resistive than silver, one square inch of 4/0 copper, for example, has a resistance of 0.88 times that of an equal area of 4/0 silver grid. This is because the copper grid contains a greater volume of

conductive metal per unit area. A cell employing 4/0 silver grid in the positive and 4/0 copper on the negative would be expected to exhibit the greatest discharge voltage.

Degree of over-pasting also affects conductivity. The same grams per square inch active material on 4/0 grids requires more overpasting than lighter grids. It is also to be noted that expanded metal grids vary considerably between manufacturers.

#### 5) Negative Material Density (Factor E)

Apparent density is important in the zinc negative for the same reasons that are associated with the positive. Efficiency of the negative active material has been observed to decrease considerably at apparent densities in excess of fifty (50) grams per cubic inch (depending somewhat upon particle size). Figure Nos. 8 and 9 correlate void space and apparent density for the zinc and divalent silver oxide plates.

#### 6) Additive Content in the Negative Plate (Factor F)

The effect of this proprietary additive is to retard hydrogen evolution on stand, thus decreasing the dissolution of zinc and reducing hydrogen liberation. This is done at some slight sacrifice of discharge voltage.

#### 7) Negative Grid Metal (Factor G)

As with positive grid metal, choice of negative grid metal is reflected in the discharge voltage. Hydrogen overvoltage is also to be considered. For example, there is a tendency for unprotected copper to corrode after long stands, resulting in loss of electrical continuity as well as copper contamination.

Figure No. 10 relates physical characteristics of the zinc plate for zinc and copper grid materials.

#### 8) Negative Formulation (Factor H)

There appeared to be little previous data relating to negative material formulation with specific emphasis on long stand, strictly primary applications.

A limited supply of "metallic" zinc, prepared by an Eagle-Picher proprietary process, was used in two test cells. This zinc is not to be confused with the standard "metallic" formulation which was studied in greater depth. It is characterized by large particle size. This resulted in unfavorable discharge voltages because of decreased active material surface area. This study was, therefore, terminated.

#### c. Mathematical Approach

As the variables for study were originally outlined (Table No. VI), more than five thousand possible combinations of cell test variables were possible.

Classical evaluation of cell variables, that is, the determination of the effect a single variable while holding all other factors constant, yields in itself, no information relative to possible interactions. In addition, this approach involves the testing of all possible combinations. A testing program of such magnitude obviously was impossible.

For this reason, the statistical approach was favored, whereby testing of a small fraction of the total possible combination permits evaluation of test variables. Table No. VIII reveals the combinations of variable levels included in the test sets of experimental cells. Testing of this type allows determination of the effect of one variable in the presence of other variables.

TABLE NO. VIII  
FRACTIONAL FACTORIAL PATTERN  
TWENTY-SEVEN CELL GROUP (1)(2)

CELL NO.	L E V E L S							
	A	B	C	D	E	F	G	H
1	0	0	0	0	0	0	0	0
2	0	1	1	1	2	1	2	2
3	0	2	2	0	1	2	1	1
4	1	0	0	0	0	1	1	1
5	1	1	1	0	2	2	0	0
6	1	2	2	1	1	0	2	2
7	2	0	0	1	0	2	2	2
8	2	1	1	0	2	0	1	1
9	2	2	2	0	1	1	0	0
10	1	0	1	1	1	0	0	1
11	1	1	2	0	0	1	2	0
12	1	2	0	0	2	2	1	2
13	3	0	1	0	1	1	1	2
14	3	1	2	1	0	2	0	1
15	3	2	0	0	2	0	2	0
16	3	0	1	0	1	2	2	0
17	3	1	2	0	0	0	1	2
18	3	2	0	1	2	1	0	1
19	2	0	2	0	2	0	0	2
20	2	1	0	0	1	1	2	1
21	2	2	1	1	0	2	1	0
22	2	0	2	1	2	1	1	0
23	2	1	0	0	1	2	0	2
24	2	2	1	0	0	0	2	1
25	0	0	2	0	2	2	2	1
26	0	1	0	1	1	0	1	0
27	0	2	1	0	0	1	0	2

- (1) Plan No. 11, p. 144, "Orthogonal Main Effect Plans", Addelman, AD 272 250
- (2) Cells including the "0" level of H were not constructed.

#### d. Cell Construction

As noted by Table No. VIII, fabrication of "pasted" negatives proved not to be practically adaptable to the primary system. (This type of plate is ordinarily in secondary cells in which the active materials are electroformed within the cell.) As remaining cells of the twenty-seven cell test group had already been constructed, it was economically unfeasible to select a new plan for combinations of cell variables. This caused, in effect, a non-orthogonal, eighteen-cell plan, including all cells not containing level "H", pasted negatives.

#### e. Cell Testing

Cells were activated with electrolyte of the appropriate strength and composition, then placed on stand at +130° F. This was done in order to accelerate the mechanisms which cause losses in capacity. Cells were removed from stand at intervals of two hours, four, six and eight days. Cells were allowed to cool to room temperature, then discharged at a thirty-ampere rate. Voltage and capacity responses were obtained for each cell and served as raw data for analysis of effects. These responses are revealed by Table Nos. IX and X.

### 3. Analysis of Data

#### a. By Examination of Means

Cell responses were categorized with respect to each variable and average responses were calculated for cells including each level of the variable. This treatment of capacity data is revealed by Table Nos. XI through XIV.

Comparison of means indicated consistent trends appeared with respect to four factors. These are summarized as follows:

- 1) Electrolyte Concentration - Factor B. Cells employing 45% KOH (B<sub>2</sub>) exhibited approximately 21% greater capacity than those having 35% KOH.
- 2) Positive Grid Metal - Factor D. Cells using nickel grid averaged 10% greater capacity than those with silver grid.
- 3) Negative Density - Factor E. Cells using the low level of Factor E (40 grams per cubic inch) averaged 13% greater capacity than those having 50 grams per cubic inch.
- 4) Negative Formulation - Factor H. Cells employing zinc of "spongy" structure exhibited capacities greater by approximately 20% than those having "metallic" zinc.

## b. Analysis by Half-Normal Plots

### 1) General

This method of analysis of factorial experiments is generally credited to Cuthbert Daniel. It is predicated on the fact that if there are no real effects or interactions in such an experiment, then the contrasts - or measures of the main effects and interactions - should be normally distributed with mean zero. This implies a linear representation of cumulative sample distribution of contrasts versus the cumulative half-normal distribution when plotted on normal probability paper. A large deviation of a contrast to the right of the half-normal line indicates either statistical significance or abnormal error.

Actual mathematical steps in applying analysis by half-normal plots are included in the Appendix.

### 2) Procedure

Calculations were first accomplished using "zero" responses for cells which failed prior to scheduled discharges. As this treatment is not indicative of progressive losses in capacity and voltage characteristics or the effect of construction variables on these gradual losses, calculations were repeated using estimated responses. These new responses were obtained by interpolating response patterns of individual cell designs. Half-normal plots derived from these data are revealed by Figure Nos. 11 through 17.

### 3) Results and Conclusions

Effects which are apparently significant with respect to capacity retention are:  $A_L$ ,  $A_C$ ,  $A_A$ ,  $B_L$ ,  $D$ ,  $E_L$ ,  $F_L$ ,  $F_A$ , and  $G_L$ .

Table No. XV relates significant effects with respect to voltage.

TABLE NO. XV

SIGNIFICANT EFFECTS  
(with respect to voltage)

2 hours	4 days	6 days	8 days	10 days
$A_C$	$A_C$	$A_C$	$A_C$	$A_Q$
$F_L$	$A_Q$	$A_Q$	$C_L$	$A_C$
$A_Q$	$E_L$	$D$	$A_Q$	$A_L$
$A_L$	$G_L$	$F_L$	$A_L$	$C_Q$
$D$	$A_L$	$A_L$	$F_L$	$G_Q$
$G_Q$	$C_L$	$E_Q$	$B_L$	$B_L$
$B_L$	$D$	$G_Q$	$D$	$D$
$C_L$	$G_Q$		$E_Q$	$B_Q$
	$B_L$		$G_Q$	$C_L$
	$F_L$			

The high apparent significance of factors relating to Factor A, electrolyte additives, stems from non-orthogonality of the plan. Six cells in each set were tested using level "A<sub>2</sub>", while the remaining levels were used in only four cells each, thus causing bias toward the "A<sub>2</sub>" level. Table No. XIX reveals mean responses for cells involving each level of Factor "A" for a stand period of six days, and is presented as representing typical date.

TABLE NO. XIX  
EFFECT OF FACTOR "A", ELECTROLYTE ADDITIVES

LEVEL OF "A"	VOLTAGE RESPONSE	CAPACITY RESPONSE
A <sub>0</sub> , no additive	82.3	17.1
A <sub>1</sub> , gel	84.8	20.3
A <sub>2</sub> , MnO <sub>2</sub>	84.9	18.4
A <sub>3</sub> , LiOH	86.1	19.3

These data indicate that, in the case of each voltage and capacity response, the cells employing no electrolyte additives exhibited the smallest responses. This implies confirmation of significance of the factors relating to "A".

### c. Curve-Fitting by Computer

#### 1) General

In order to achieve the maximum benefit from the response data previously discussed, it was decided to derive a formula describing voltage and capacity data as a function of cell construction variables.

#### 2) Procedure

The equation is of the type

$$Y = \mu + a_i + B_1b + B_2b^2 + \gamma_1c + \gamma_2c^2 + d_j + \delta_1e + \delta_2e^2 + K_1f + K_2f^2 + g_k + h_e. \quad (1)$$

The letters b, c, e and f refer to the variable level numbers employed in a certain cell (see Table No. VIII). The terms a<sub>i</sub>, d<sub>j</sub>, g<sub>k</sub> and h<sub>e</sub> represent values calculated by computer to indicate the effect of levels of the qualitative Factors A, D, G and H. The terms μ, B<sub>1</sub>, B<sub>2</sub>, γ<sub>1</sub>, γ<sub>2</sub>, δ<sub>1</sub>, δ<sub>2</sub>, K<sub>1</sub> and K<sub>2</sub> were also determined by computer by the method of least squares.



Values were determined for all terms included in equation (1) to allow description of both capacity and voltage responses for stand periods of two hours, four days, six days, eight days and ten days at a temperature of +130° F. Table No. XX reveals values used in calculating responses.

### 3) Results

The experimental series included testing of only a fraction of the many possible cell designs. It was anticipated that if effects of the test variables were consistent and included no important interactions, equation (1) might satisfactorily predict responses for any of the untested designs. This proved to be impractical, however. For instance, after a stand period of eight days at +130° F, Cell Nos. 19 and 25 were internally shorted. Therefore, terms of the least squares equation were determined from sixteen rather than eighteen responses. Table No. XXI contains comparative data whereby the response deterioration characteristics were used to closely estimate responses which Cell Nos. 19 and 25 might have exhibited had they undergone normal performance degradation.

These calculated responses are indicated by the points falling away from the response curves in Figure Nos. 18 and 19.

### 4) Conclusions

It is indicated that there may be interactions between several of the cell variables since the effect of including a specific design factor is not predictably constant among a large number of cell design combinations. Such interactions would require a larger orthogonal fractional replicate test group for complete evaluation. Data in Figure Nos. 18 and 19 also re-emphasize another fundamental fact. Direct comparison of the data comprising these figures reveals that several cells representing maxima on the capacity response pattern correspond to minima on the voltage response pattern. Slopes of corresponding straight line segments represented by the two figures are frequently of opposite algebraic sign. This again confirms that measures commonly taken to improve capacity retention are deleterious to voltage control, and vice versa. This indicates the difficulty encountered in producing an optimum "general purpose" cell design. Data of Figure Nos. 18 and 19 do, however, indicate that Cell Nos. 10 and 14, following eight days activated stand at +130° F, exhibit the greatest voltage responses, while their capacity responses are also well above the mean. Many features of these cells have been included in the design of the pre-prototype and prototype cells.

## F. Phase IV - Pre-Prototype and Prototype Cell Design

### 1. Objective

It was noted, in association with the fractional factorial study, that cells employing nickel positive grids exhibited greater retention-of-charge during stand periods up to ten days at +130° F. It was further indicated that at stand periods greater than ten days, this trend might tend to reverse, with cells having nickel positive grids suffering total failure first. To study this possibility and evaluate a cell design using other information gained in the fractional factorial study, a series of pre-prototype cells was constructed.

## 2. Cell Construction and Testing

An equal number of cells were constructed using each nickel and silver positive grid metal. Other construction variables were as follows:

- a. Electrolyte - 45%, no additives
- b. Positive material density - 80 gram per cubic inch
- c. Negative material density - 40 gram per cubic inch
- d. Negative grid metal - 4/0 expanded copper
- e. Negative material additive - 2%
- f. Negative formulation - spongy zinc.

Cells were placed on stand at +130° F and open circuit voltages recorded periodically. These are displayed by Table Nos. XXIII and XXIV. Cells were removed from stand in numerical sequence and discharged at 30 amperes to preselected stand periods of three, five, seven, eleven and seventeen days.

Discharge characteristics for cells on stand test are displayed by Figure Nos. 20 through 24.

Data in Figure No. 25 reveals a significant variance between open circuit voltage retention characteristics of the two experimental series of cells. Cells having silver positive grid metal maintained higher open circuit voltage. In addition, following seventeen days activated stand, none of the remaining five cells having nickel grid yielded useable capacity, while cells having silver positive grid yielded only slightly decreased voltage. Increased open circuit voltage exhibited by cells having silver positive grid metal is apparently reflected in the initial voltage during the first few minutes on discharge. Cells employing nickel positive grids display slightly greater ampere-hour efficiency. However, capacities of the two cell types converge with increased activated stand periods and cells employing nickel positive grids were first to suffer total failure. Further studies would be necessary to determine the minimum silver grid which may be used without sacrificing voltage control during high rate discharges.

## 3. Prototype Cell Design

The following cell design has been selected as a result of stand tests of experimental cells at elevated temperature:

- a. Positive material - 1.8 gms/in<sup>2</sup> of AgO
- b. Positive grid metal - silver
- c. Positive material apparent density - 80 gms/cu in
- d. Negative material formulation - spongy zinc\*
- e. Negative material plate weight - 1.35 gms/in<sup>2</sup>
- f. Negative grid material - copper
- g. Negative material apparent density - 40 gms/cu in
- h. Separation (1) one wrap woven nylon  
(2) two wraps cellulosic membrane  
(3) one wrap non-woven rayon

\* This is a descriptive name applied to dry-charged spongy zinc active material prepared by an Eagle-Picher proprietary process.

- i. Electrolyte - 42 to 45% KOH by weight
- j. Plate size - 1-5/8 in. x 2-1/8 in.
- k. Number of plates (1) seven positive plates  
(2) eight negative plates.

This design differs from the previous control design in the following design factors:

- a. Positive grid metal (silver vs. nickel)
- b. Negative material density (decreased density is suggested)
- c. Type of separation (multiple wraps on separation to be used next to the positive plate)
- d. Strength and composition of electrolyte (more concentrated electrolyte)
- e. Plate size (height has been increased by 1/8 in.)

Evaluation of individual cells of improved design has indicated general limits of satisfactory activated stand. For instance, no cells were observed to have failed after a stand period of four days at +130° F. It is indicated that individual cells may be expected to perform reliably after a minimum of four days stand at +130° F. An activated life of two weeks appears obtainable at a stand temperature of +75° F. These improvements in overall cell performance result from changes in cell design and manufacturing processes as well as greater quality assurance measures. That is, the activated stand life of a primary cell has been significantly increased at no appreciable loss in energy density.

#### IV. SUMMARY AND CONCLUSIONS

Studies categorized under four phases have been conducted resulting in an improved primary zinc-silver oxide cell.

1. Commercially available separator materials, both membranous and "open", were evaluated with respect to electrical resistance, speed of wetting and absorptivity (electrolyte retention). In addition, life cycle tests indicated relative abilities of separators to withstand conditions of service. Cellulosic products have the advantage of low resistance which is necessary for high rate applications.

2. A preliminary cell design study consisted of stand tests of cells constructed using four distinct separator combinations, including ion-exchange membranes. It was indicated that a considerable percentage of individual cells, if constructed by the most rigorously controlled processes, might perform satisfactorily after stand periods of weeks at room temperature. The most desirable electrical characteristics were obtained from cells employing multiple wraps of a cellulosic membrane in combination with a woven nylon fabric. The "open" separator removes the cellulosic material from the oxidizing effect of the divalent silver, while providing a region in which crystalline growths related to the positive plate can occur with reduced danger of damage to the cellophane membrane. This separation technique was selected for use in the following evaluation of other cell construction variable studies.

3. A statistically designed experiment was outlined to study the effects of several variables, including electrolyte strength and composition, positive and negative material densities, positive and negative grid metals, and negative formulation.

Duplicate sets of eighteen different cell designs were discharged following activated stand periods of two hours, four, six, eight and ten days at +130° F. Several thousand cells would have been necessary to test these variables by the "classical" method of varying only one factor at a time. Response data were analyzed by simple examination of means, as well as by preparation of half-normal plots.

Consistently beneficial effects upon capacity retention resulted from more concentrated KOH electrolyte (Level B<sub>2</sub>), less dense negative material (Level E), nickel positive grid (Level D) and "sponge" negative material (Level H<sub>1</sub>). Less definitive results include use of electrolyte additives, such as LiOH, MnO<sub>2</sub> and PVA gel.

Important desirable effects upon voltage were: use of silver positive grid (as, of course, anticipated), and use of less dense negative material.

Analysis by half-normal plots is essentially a conservative method which is not advantageous in locating effects of only minor magnitude or significance. Mathematical computations are greatly complicated by non-orthogonal plans and may be too laborious to accomplish manually.

4. A series of pre-prototype cells were designed, constructed and tested. These cells, the construction details of which have been presented, utilized knowledge gained in the three previous phases of study. Cells were placed on activated stand at +130° F to speed failure mechanisms. Equal numbers of cells were constructed using silver and nickel positive grid metals to gain further insight into the relative performance characteristics, particularly following stand periods greater than ten days. There were no other construction variables.

Data revealed slightly greater capacity efficiency initially for cells having nickel positive grids, although the difference became less significant following several days activated stand. Cells having nickel positive grids were first to suffer internal shorting.

Although reliability studies as such were not conducted, it is apparent that a large percentage of individual cells could be expected to perform satisfactorily following four days activated stand at +130° F or approximately two weeks at +75° F.

Because of activating heating in batteries and the number of cells involved, battery reliability would be expected to be less than the above limits. It would be unwise to attempt to apply single cell capacity retention data to produce an estimate of battery reliability.

V. PERSONNEL

The following totals of man-hours have been expended during the contract period:

Engineering	- 1549 hours
Technical	- <u>2542 hours</u>
TOTAL	- 4091 hours

BRH/bk

A P P E N D I X

# MASTER SCHEDULE

PRIMARY ZINC-SILVER OXIDE BATTERY

NAS 8-5493

FA-242

1 July 1963

P.O. DATE

PROGRAM

CONTRACT

REFERENCE

PREPARED

APPROVED

APPROVED

APPROVED

MILESTONES

## ACTIVITIES

### SEPARATOR STUDIES

- Open Types
- Closed Types
- Special Types

### ACTIVE MATERIAL FORMULATION

- Positive Material
- Negative Material

### PLATE CONSTRUCTION AND

#### ELEMENT DESIGN

- Spongy vs. Metallic Zinc
- Dry Charged Spongy Zinc
- Grid Materials

### ELECTROLYTE

- Effect of Concentration
- Additives to Electrolyte

### CELL AND BATTERY CONSTRUCTION

- Electrolyte Retention
- Voltage Regulation
- Energy Density
- Thermal Characteristics
- Evaluation of State-of-Art

### REACTION MECHANISMS

- Theoretical Investigation
- Gassing Rate

### RETENTION EVALUATION

### PROGRESS REPORTS

\* Monthly Progress Report

X Pre-prototype cell construction

O Prototype cell construction

Schedule

Progress

Revision

AFTER GO-AHEAD

CALENDAR

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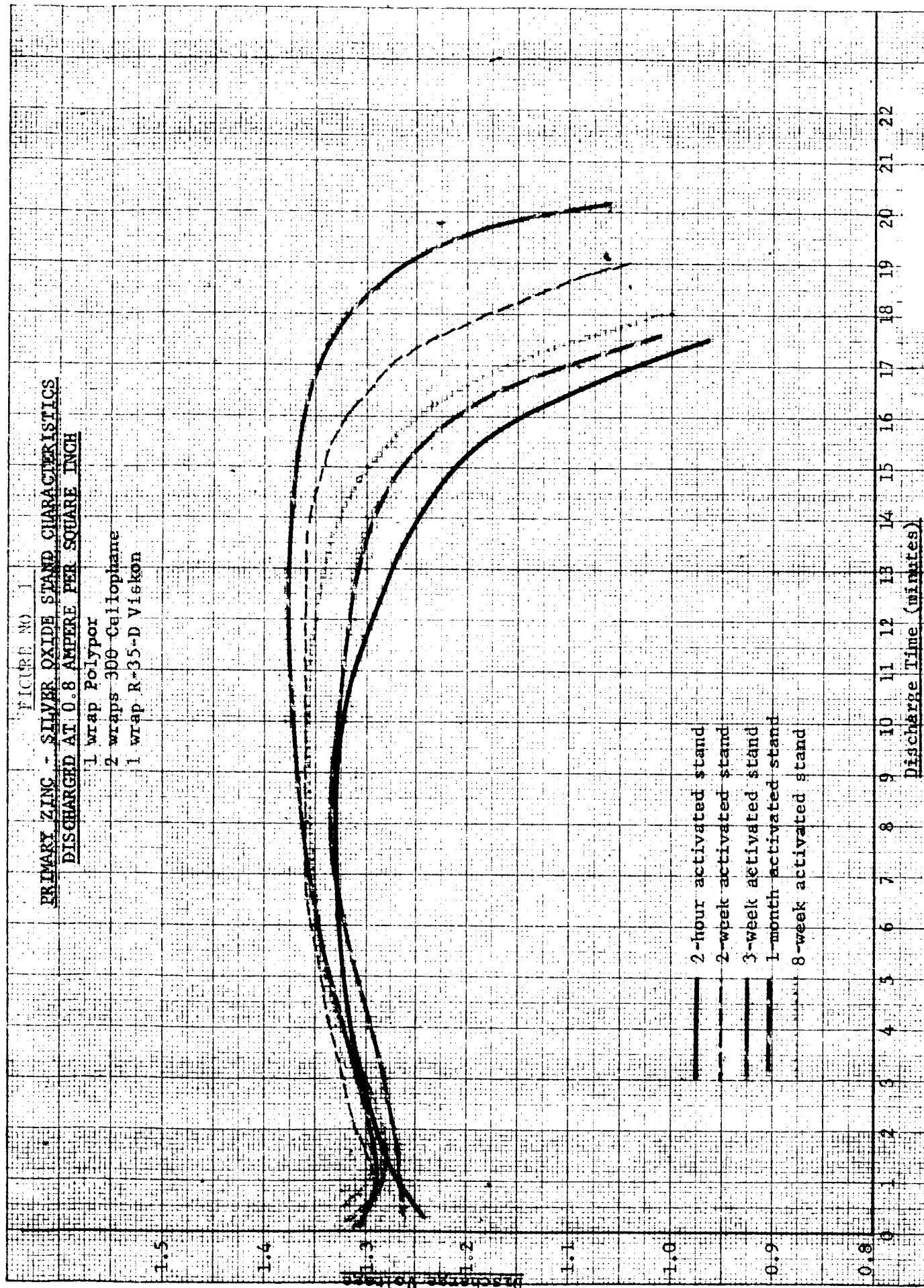
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FIGURE NO. 1

PRIMARY ZINC - SILVER OXIDE STAND CHARACTERISTICS  
DISCHARGED AT 0.8 AMPERE PER SQUARE INCH

- 1 wrap Polypor
- 2 wraps 300 Cellophane
- 1 wrap R-35-D Viskon



**FIGURE NO. 2**  
**PRIMARY ZINC - SILVER OXIDE STAND CHARACTERISTICS**  
**DISCHARGED AT 0.8 AMPERE PER SQUARE INCH**  
 3 wraps Pb film  
 1 wrap R-35-D Viskon

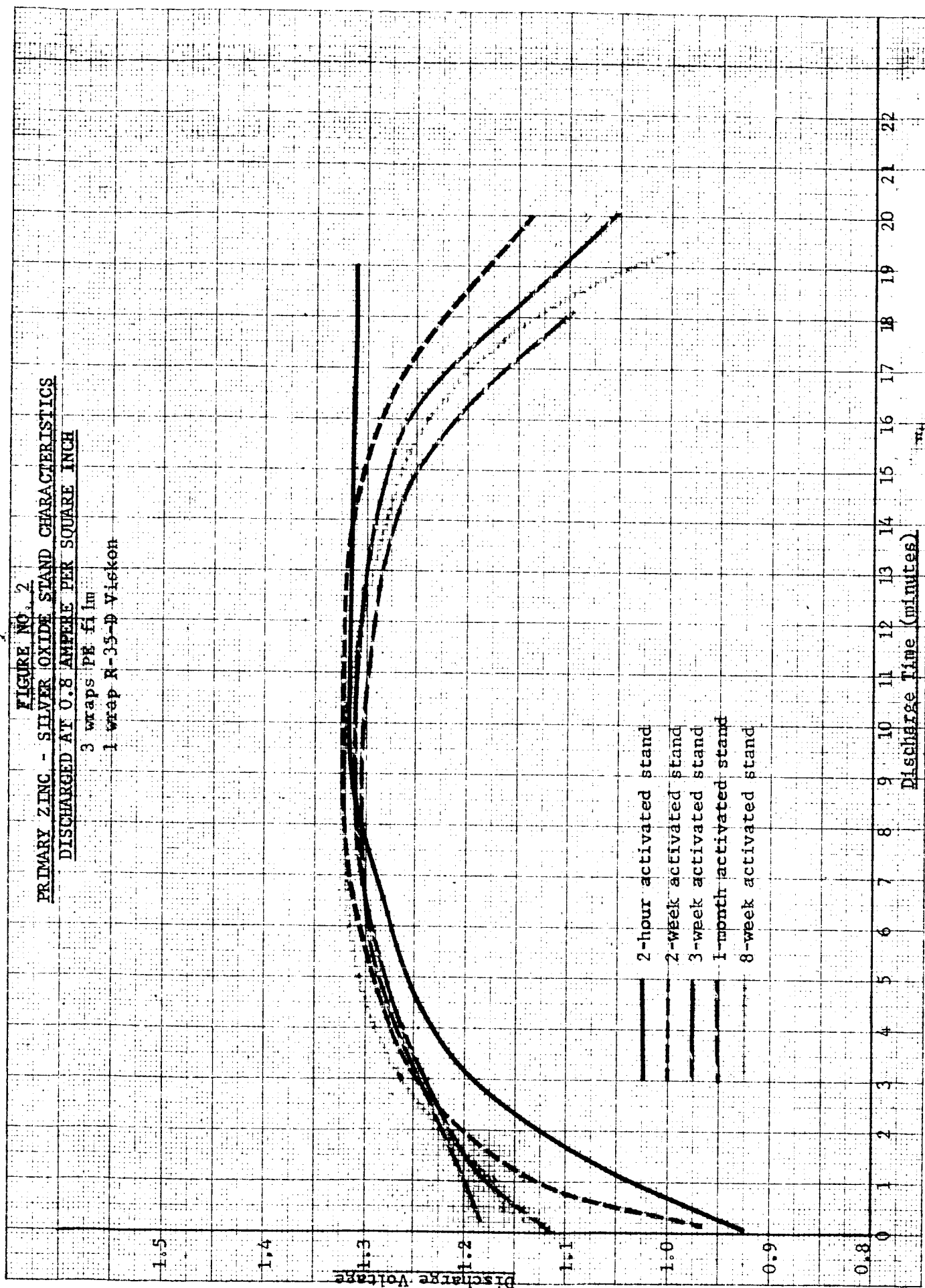


FIGURE NO. 3

PRIMARY ZINC - SILVER OXIDE STAND CHARACTERISTICS

DISCHARGED AT 0.8 AMPERE PER SQUARE INCH

- 1 wrap Nylon
- 2 wraps 300 Cellophane
- 1 wrap R-35-D Viskon

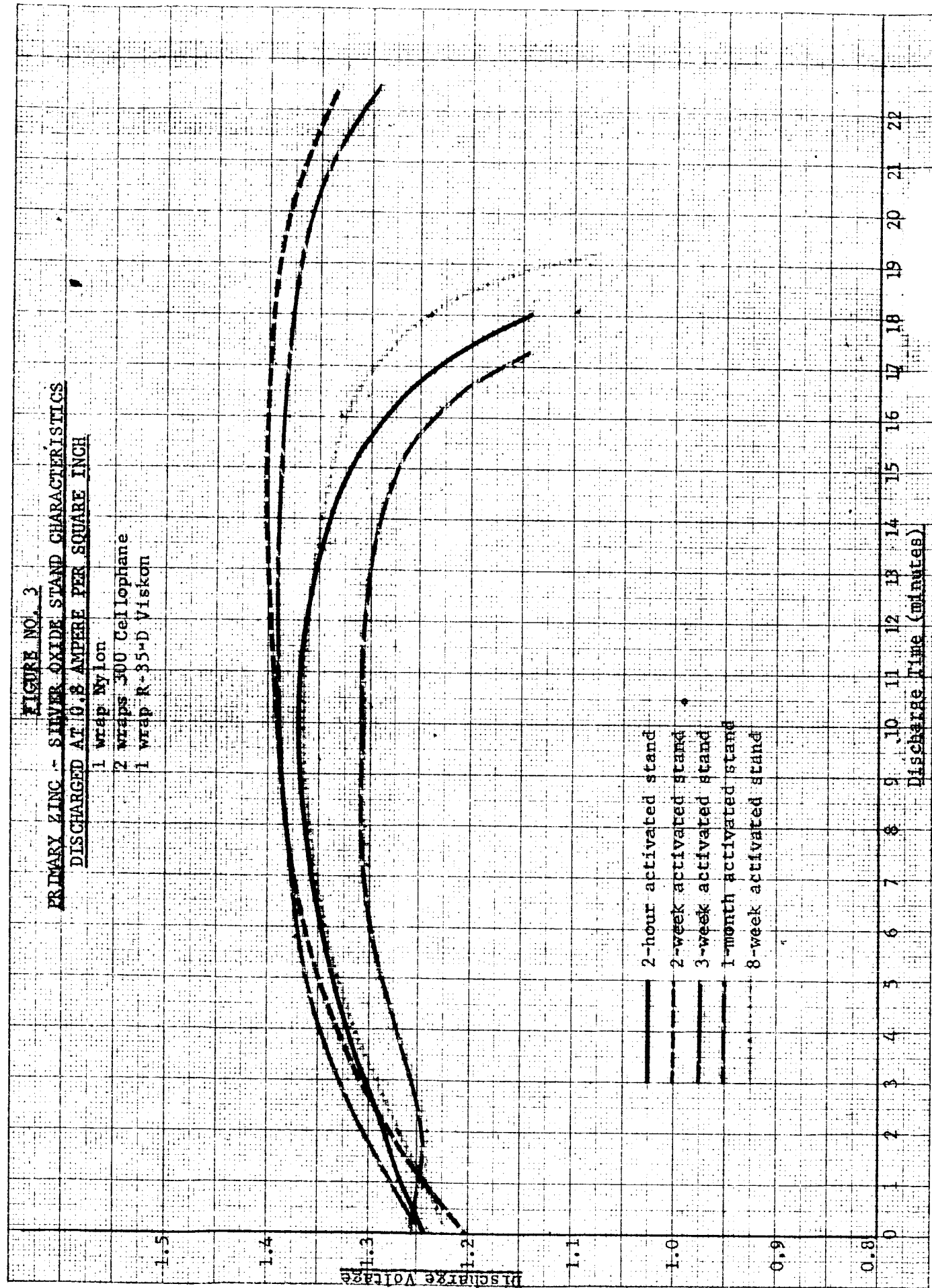


FIGURE NO. 4  
 PRIMARY ZINC-SILVER OXIDE STAND CHARACTERISTICS

Discharged at 0.8 amp./sq.in.

- 1 wrap 133 Visking
- 1 wrap R-75-D Viskon

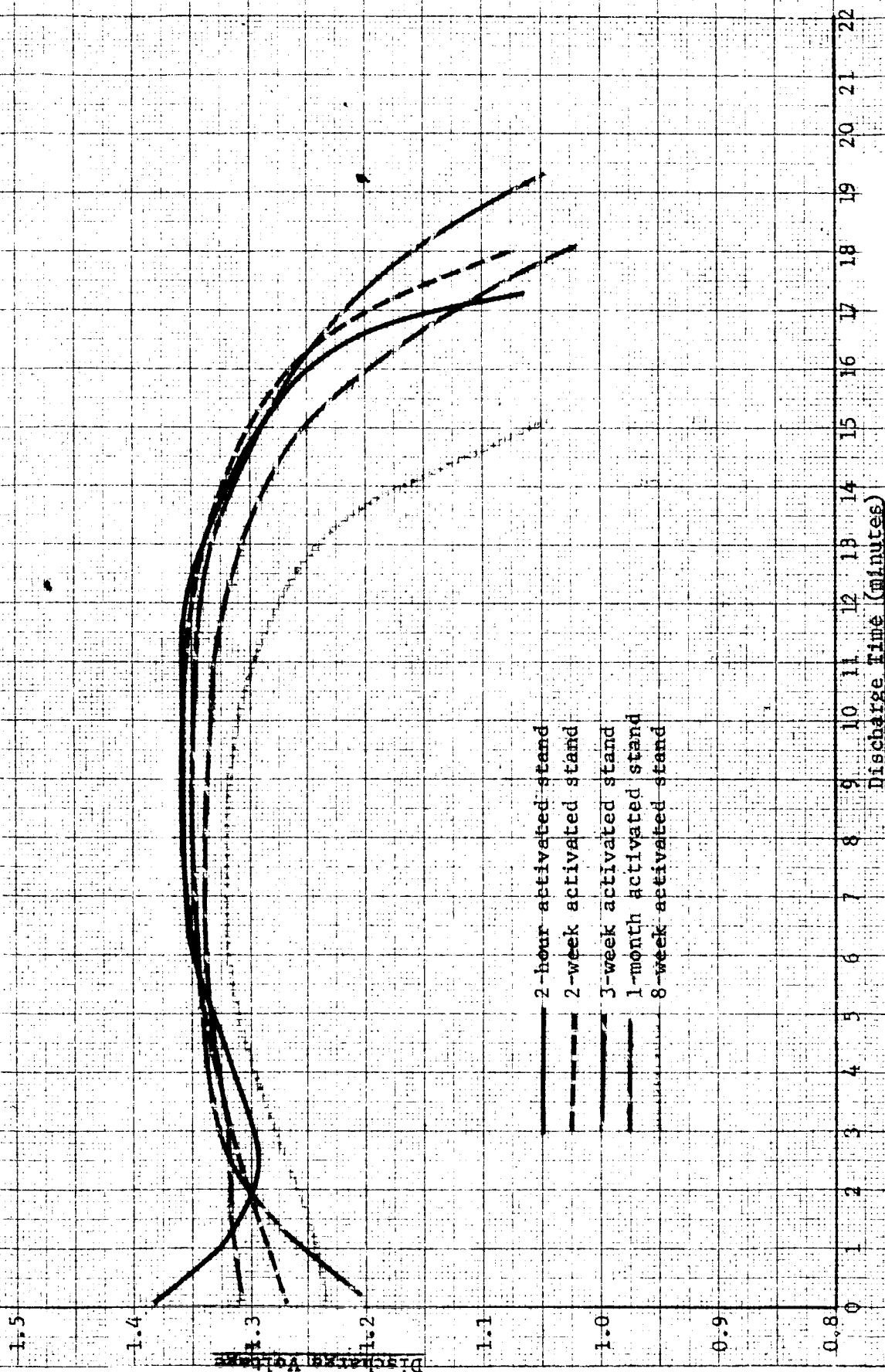


FIGURE NO. 5

EFFECT OF STAND ON CELL CAPACITY  
Discharged at 0.8 amp./sq.in.

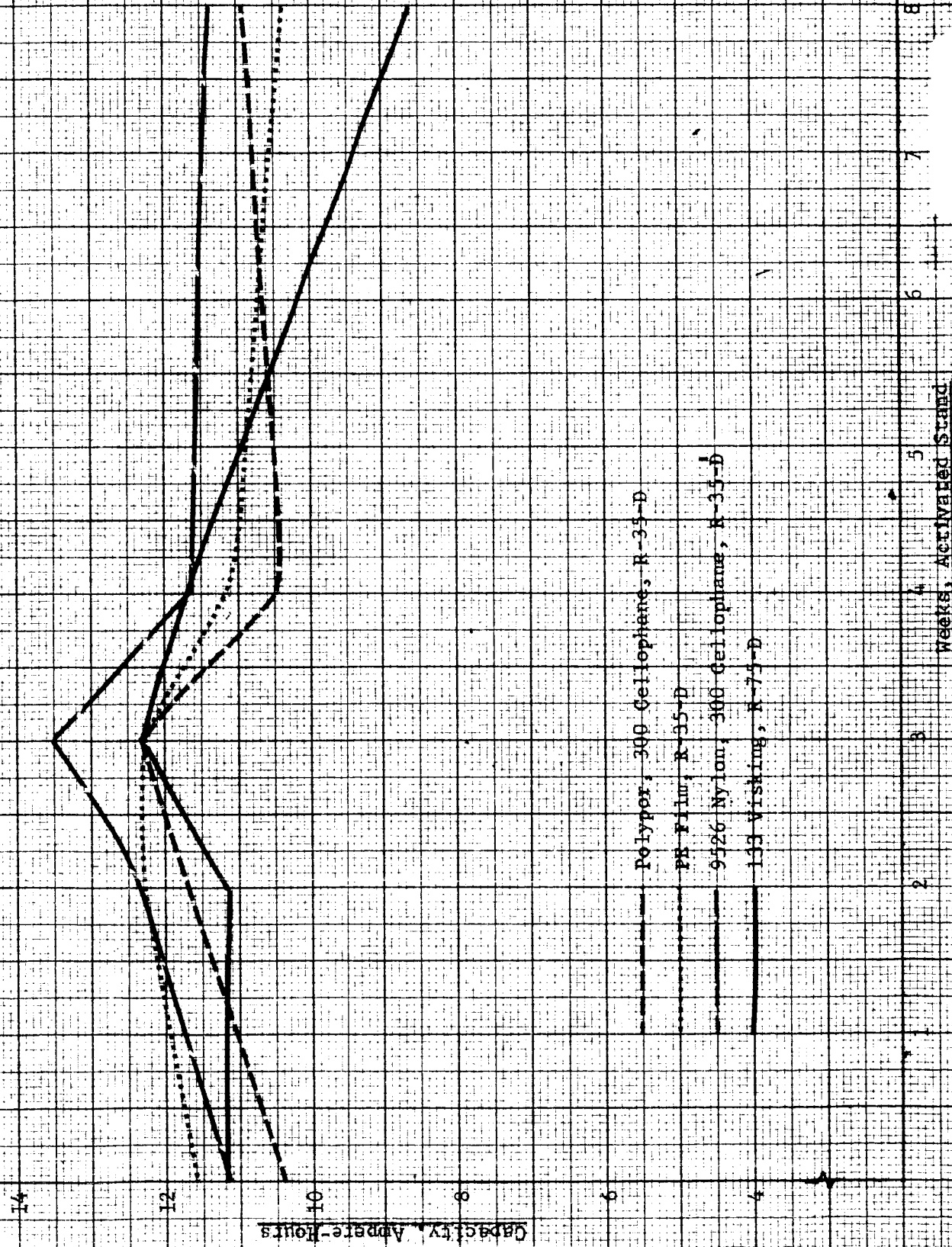


TABLE NO. III

SEPARATOR LIFE CYCLE STUDY  
(Cycles to Failure)

CELL NO.	NUMBER WRAPS	SEPARATOR COMBINATION	CYCLES TO FAILURE
1	1	9526 Nylon .002" Polyvinyl Alcohol Film	879
2	2	.002" Polyvinyl Alcohol Film	347
3	1 2	9526 Nylon 300 Cellophane	347
4	1 1 1	9526 Nylon 300 Cellophane .002" Polyvinyl Alcohol Film	347
5	1 ea	9526 Nylon, 600 Cellophane	347
6	1 ea	Dynel, Fibrous Visking	347
7	1 ea	Dynel, 133 Visking	347
8	1 1	9526 Nylon 300 Cellophane coated with PVA on one side	347
9	1 1	9526 Nylon .002" Polyvinyl Alcohol Film	171
10	1	.002" Polyvinyl Alcohol Film	144
11	1 1	9526 Nylon .004" Polyvinyl Alcohol Film	749
12	1 3	9526 Nylon PE 10/20 Film	347
13	1 ea	Dynel, Polypor, Polyvinyl Alcohol Film	879
14	1 3	9526 Nylon PE 10/20 Film	1232
15	1 2	9526 Nylon PE 10/20 Film	347
16	1 ea	9526 Nylon, 300 Cellophane and Polyvinyl Alcohol Film	1232
17	1 ea	PE Film (special), 9526 Nylon	528
18	2 1	PE Film (special) 9526 Nylon	528

TABLE NO. IV  
EFFECT OF TEMPERATURE  
ON SEPARATION PROPERTIES

SEPARATOR MATERIAL	RESISTANCE (ohm-inch <sup>2</sup> )			TR <sub>2</sub> (minutes)**		
	0° F	+80° F	+120° F	0° F	+80° F	+120° F
133 Visking	0.054	0.0225	0.011	30	4	4
Fibrous Visking		0.0143			2	
300 Cellophane	0.0269	0.0053	*	1.75	0.5	+
600 Cellophane	0.025	0.0116	0.00718	4	0.5	0.5
PE Film-307	0.0898	0.0071	*	2	1	+
PVA Film	0.0322	0.0071	0.0071	20	3	1.2
0.005" Nylon		0.0024			+	
0.011" Nylon		0.0055			+	
0.005" Pellon		0.0071	0.0071		+	1 min.
0.012" Pellon	0.0269	0.0116	0.00359	0.55	+	1 min.
R-35-D	0.00898	0.0084	*	1	+	0.85
R-75-D	0.00898	0.0112	0.00718	2	1	+

\* Resistance too small to be detected.

\*\* TR<sub>2</sub> represents the soak time required for the sample to attain a resistance equal to twice its final stable value.

+ Resistance reached final almost immediately.

Blank spaces indicate data not yet obtained.

TABLE NO. V

CHARACTERISTICS OF VARIOUS SEPARATOR MATERIALS  
(one layer at +80° F - 72-hour soak)

SEPARATOR MATERIAL	DIMENSIONS (in.)	AREA (sq. in.)	DRY WEIGHT (gms.)	WET WEIGHT (gms.)	ELECTROLYTE RETENTION BASIS-1 in <sup>2</sup>		ABSORBENCY $\frac{\text{Wt. Wet} - \text{Wt. Dry}}{\text{Wt. Dry}}$	THICKNESS, WET (in.)	RESISTANCE	
					Gm. KOH	cc KOH			ohm-in <sup>2</sup>	ohm-in*
PE Film-307	12.0 x 12.0	144	3.1138	11.0750	0.0552	0.0423	2.56	0.002	0.0071	3.05
PVA Film	6.25 x 12.0	75	2.5428	7.4120	0.00649	0.0499	1.84	0.002	0.0071	3.05
300 Cellophane	6.0 x 12.0	72	1.6628	7.6100	0.0826	0.0635	3.58	0.003	0.0053	1.76
600 Cellophane	11.0 x 11.0	121	4.2462	18.0970	0.1140	0.0880	3.26	0.0042	0.0116	2.52
Visking 133	5.0 x 6.0	30	2.1830	7.4130	0.1743	0.1340	1.94	0.009	0.0225	2.52
Fibrous Visking	5.0 x 6.0	30	1.7290	6.1970	0.1489	0.1145	2.52	0.009	0.0143	1.59
R-35-D	6.0 x 6.0	36	0.6517	7.0190	0.1768	0.1360	7.96	0.00575	0.0084	1.46
R-75-D	2.5 x 6.5	16.25	0.6120	5.7050	0.3134	0.2410	8.33	0.0105	0.0112	1.07
0.003" Nylon	2.5 x 7.0	17.5	0.2580	1.0384	0.0445	0.0342	3.03	0.003	0.0014	0.464
0.005" Nylon	12.0 x 12.0	144	4.3434	10.4103	0.0421	0.0323	1.400	0.005	0.0024	0.480
0.011" Nylon	2.63 x 6.5	16.01	0.6561	1.5260	0.0541	0.0416	1.305	0.011	0.0055	0.500
0.005" Pellon	10.0 x 10.0	100	3.6164	10.2160	0.0659	0.0507	1.83	0.005	0.0071	1.420
0.011" Pellon	6.0 x 6.0	36	1.3800	12.8850	0.3195	0.2458	8.33	0.011	0.0116	1.055
Polypor	9.0 x 9.0	81	2.9514	10.3075	0.0908	0.0698	1.83	0.00425	0.00826	1.94

\* This value is obtained by dividing the resistance in the preceding column (ohm-inch<sup>2</sup>) by the wet thickness in inches.



FIGURE NO. 8  
 VOID SPACE IN THE ZINC PLATE  
 AS AFFECTED BY APPARENT DENSITY

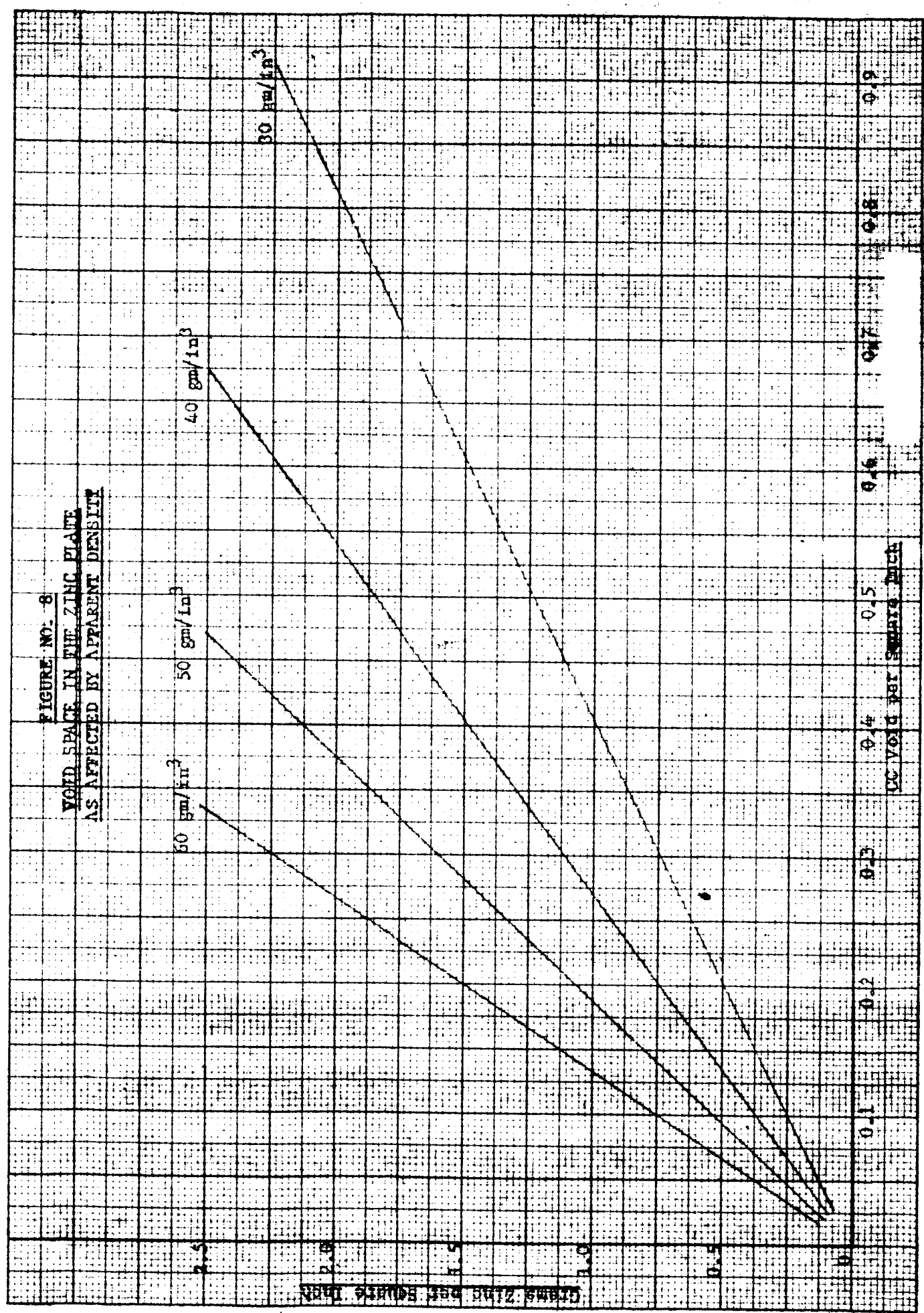


FIGURE NO. 9

VOID SPACE IN THE DIVALENT SILVER OXIDE PLATE  
AS AFFECTED BY APPARENT DENSITY

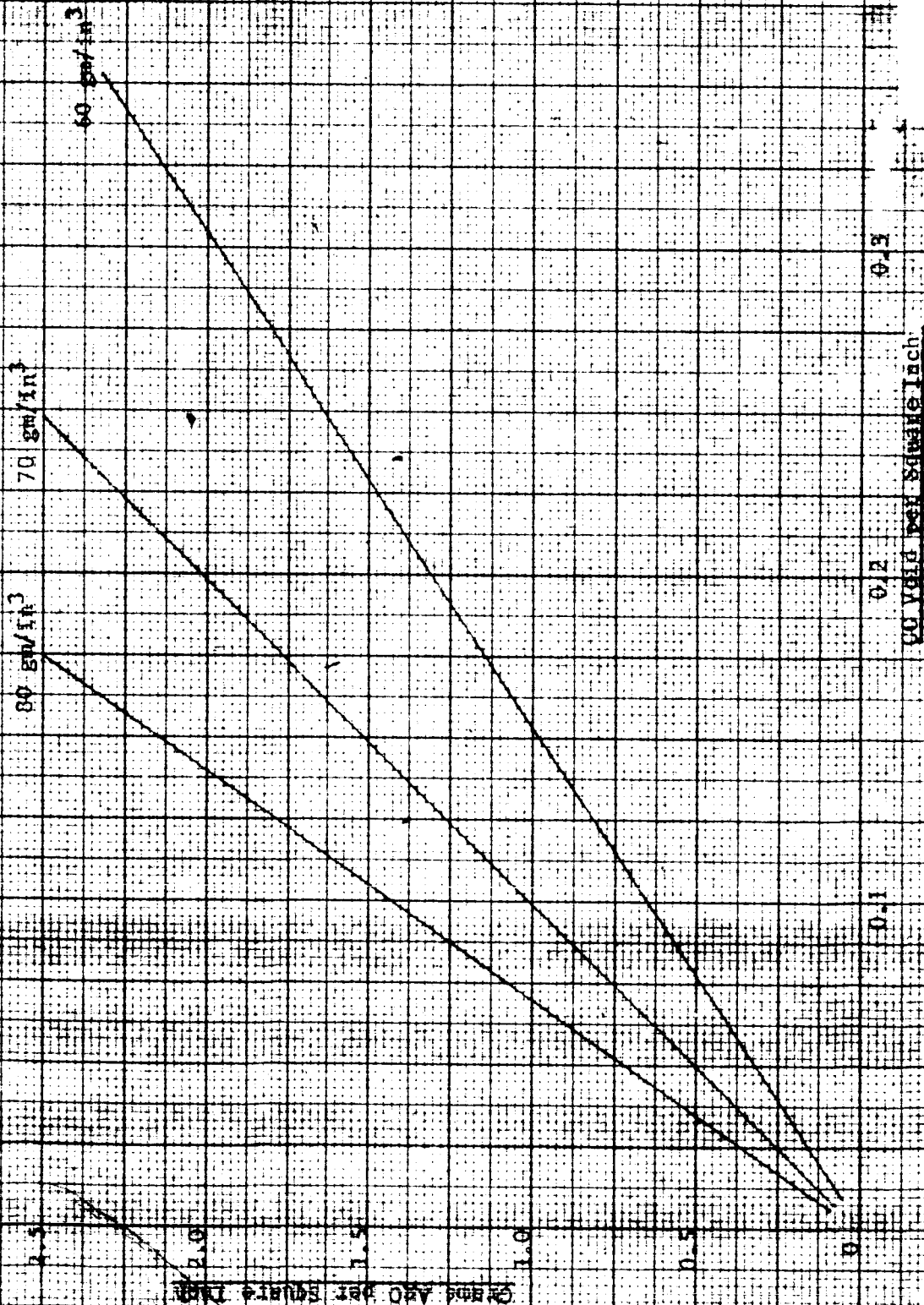


FIGURE NO. 10

THICKNESS OF THE ZINC PLATE

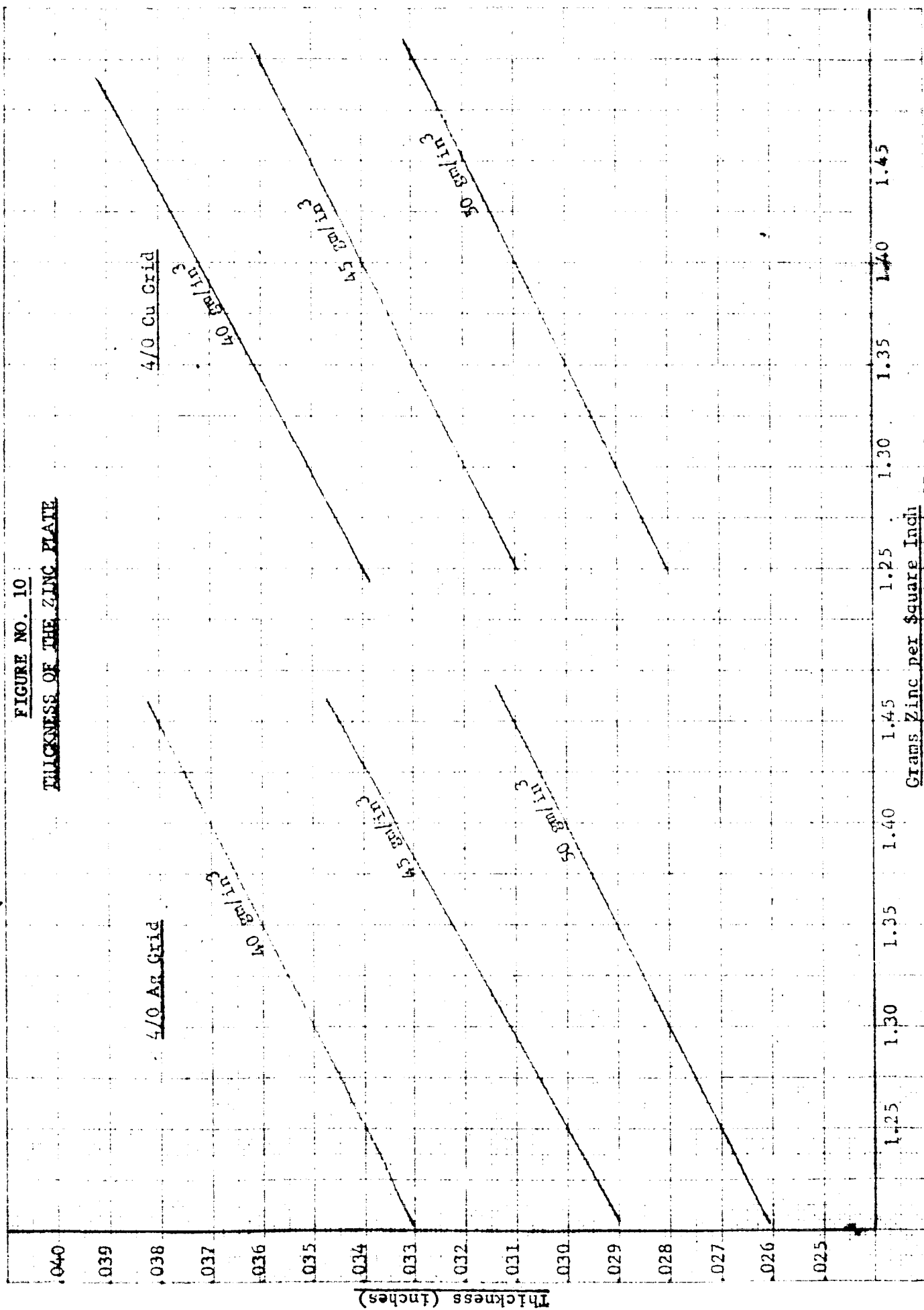


TABLE NO. IX

CELL RESPONSES (capacity)  
(Discharge capacity in minutes at a 30-ampere rate)

CELL NO.	2 hr.	4 da.	6 da.	8 da.	10 da.
2	19.0	13.0	12.25	11.0	9.0
3	30.0	28.0	23.00	21.0	18.0
4	27.0	24.0	21.00**	19.0	19.5
6	24.0	20.0	19.5	17.0	19.0
7	22.0	17.0	13.75	14.0	15.0
8	29.0	23.0	22.50	18.0	17.0
10	24.0	22.0	21.20	20.0	18.0
12	28.5	23.0	19.25	15.5	19.0
13	22.0	13.0	17.30	9.0	9.0
14	25.5	21.0	20.75	20.0	18.0
17	27.0	25.0	21.50	19.0	17.0
18	24.0	19.0	19.75	17.0	16.0
19	23.0	20.0*	11.50	14.0*	19.0
20	30.5	24.0*	19.75	19.0	20.0
23	25.0	28.0	18.50	17.0	17.0
24	28.0	25.0	24.50	22.5	22.0
25	24.0	13.0	15.50	13.0*	12.0*
27	30.0	21.0	17.50	20.0	17.0*

\*These are estimated responses based upon trends in  
cell performance characteristics.

TABLE NO. X

RESPONSES (voltage\*\*)

CELL NO.	2 hr.	4 da.	6 da.	8 da.	10 da.
2	96.0	86.0	85.3	84.0	84.0
3	88.7	78.7	81.3	73.3	76.0
4	92.7	86.0	85.0*	84.0	84.7
6	94.7	89.3	86.0	80.7	82.7
7	96.0	92.0	38.0	85.3	88.7
8	90.7	79.3	82.0	78.7	83.3
10	96.0	92.0	92.0	88.0	89.3
12	90.7	76.0	76.0	74.0	79.3
13	90.7	84.7	85.3	82.0	86.0
14	94.7	95.3	92.0	89.3	89.3
17	87.3	77.3	82.0	73.3	82.0
18	93.3	85.3	85.3	74.7	77.3
19	90.7	88.0*	84.0	80.0*	77.3
20	91.3	84.0*	81.3	77.3	82.0
23	92.0	80.7	85.3	80.0	82.7
24	89.3	76.0	80.7	75.3	79.3
25	92.0	84.7	86.7	84.0*	82.0*
27	90.7	79.3	79.3	76.3	75.0*

\* Responses for these cells (which failed to deliver usable capacity) were estimated from the response pattern.

\*\* These responses are obtained by dividing the cell discharge voltage after 15 seconds by 1.50 volts. This artificial response facilitates handling of data.

TABLE NO. XI

MEAN CAPACITY IN MINUTES  
(30-ampere rate)  
(2 hr. stand)

FACTOR	<u>L E V E L</u>			
	0	1	2	3
A	25.8	26.0	26.3	24.6
B	23.5	26.0	27.4	
C	26.1	25.3	25.6	
D	27.0	23.1	--	
E	26.6	25.9	24.6	
F	25.8	24.8	25.8	
G	25.3	27.3	24.6	
H		26.9	24.5	

TABLE NO. XII

MEAN CAPACITY IN MINUTES  
(30-ampere rate)  
(6 da. stand)

FACTOR	<u>L E V E L</u>			
	0	1	2	3
A	17.1	20.0	18.6	19.8
B	15.1	19.2	20.6	
C	18.2	19.2	18.6	
D	19.2	17.9		
E	19.6	19.9	16.8	
F	20.1	17.3	18.9	
G	18.2	20.6	17.5	
H	--	20.9	16.8	

TABLE NO. XIII

MEAN CAPACITY IN MINUTES  
(30-ampere rate)  
(8 da. stand)

FACTOR	<u>L E V E L</u>			
	0	1	2	3
A	17.1	17.9	18.1	16.2
B	17.1	17.3	18.7	
C	16.9	16.0	19.2	
D	16.6	16.5		
E	19.1	17.2	15.4	
F	19.3	15.8	17.5	
G	17.9	16.9	16.7	
H	--	19.6	15.3	

TABLE NO. XIV

MEAN CAPACITY IN MINUTES  
(30-ampere rate)  
(10 da. stand)

FACTOR	<u>L E V E L</u>			
	0	1	2	3
A	13.5	18.9	18.5	15.0
B	16.1	16.3	18.8	
C	17.7	15.0	18.2	
D	17.8	15.8		
E	18.3	16.8	16.0	
F	18.7	14.7	17.4	
G	17.2	16.6	17.0	
H	--	18.9	15.5	

TABLE NO. XVI

## FACTOR LEVELS AND COMPARISONS

CELL NO.	FACTOR LEVELS								COMPARISONS *														
	A	B	C	D	E	F	G	H	A <sub>L</sub>	A <sub>C</sub>	A <sub>Q</sub>	B <sub>L</sub>	B <sub>Q</sub>	C <sub>L</sub>	C <sub>Q</sub>	D**	E <sub>L</sub>	E <sub>Q</sub>	F <sub>L</sub>	F <sub>Q</sub>	G <sub>L</sub>	G <sub>Q</sub>	H
2	0	1	1	1	2	1	2	2	-3	-1	1	0	2	0	2	2	1	-1	0	2	1	-1	1
3	0	2	2	0	1	2	1	1	-3	-1	1	1	-1	1	-1	-1	0	2	1	-1	0	2	-1
4	1	0	0	0	1	1	1	1	-1	3	-1	-1	-1	-1	-1	-1	-1	-1	0	2	0	2	-1
6	1	2	2	1	1	0	2	2	-1	3	-1	1	-1	1	-1	2	0	2	-1	-1	1	-1	1
7	2	0	0	1	0	2	2	2	1	-3	-1	-1	-1	-1	-1	2	-1	-1	1	-1	1	-1	1
8	2	1	1	0	2	0	1	1	1	-3	-1	0	2	0	2	-1	1	-1	-1	-1	0	2	-1
10	1	0	1	1	1	0	0	1	-1	3	-1	-1	-1	0	2	2	0	2	-1	-1	-1	-1	-1
12	1	2	0	0	2	2	1	2	-1	3	-1	1	-1	-1	-1	-1	1	-1	1	-1	0	2	1
13	3	0	1	0	1	1	1	2	3	1	1	-1	-1	0	2	-1	0	2	0	2	0	2	1
14	3	1	2	1	0	2	0	1	3	1	1	0	2	1	-1	2	-1	-1	1	-1	-1	-1	1
17	3	1	2	0	0	0	1	2	3	1	1	0	2	1	-1	-1	-1	-1	-1	-1	0	2	1
18	3	2	0	1	2	1	0	1	3	1	1	1	-1	-1	-1	2	1	-1	0	2	-1	-1	-1
19	2	0	2	0	2	0	0	2	1	-3	-1	-1	-1	1	-1	-1	1	-1	-1	-1	-1	-1	1
20	2	1	0	0	1	1	2	1	1	-3	-1	0	2	-1	-1	-1	0	2	0	2	1	-1	-1
23	2	1	0	0	1	2	0	2	1	-3	-1	0	2	-1	-1	-1	0	2	1	-1	-1	-1	1
24	2	2	1	0	0	0	2	1	1	-3	-1	1	-1	0	2	-1	-1	-1	-1	-1	1	-1	-1
25	0	0	2	0	2	2	2	1	-3	-1	1	-1	-1	1	-1	-1	1	-1	1	-1	1	-1	-1
27	0	2	1	0	0	1	0	2	-3	-1	1	1	-1	0	2	-1	-1	-1	0	2	-1	-1	1
Σc <sup>2</sup>									82	98	18	12	36	12	36	36	12	36	12	36	12	36	18
√ Σc <sup>2</sup>									9.05	9.90	4.24	3.464	6.0	3.464	6.0	6.0	3.464	6.0	3.464	6.0	3.464	6.0	4.24

\* The signed values displayed in the columns below are determined on the basis of the factor levels incorporated in a specific cell, and the comparison to be studied, whether linear, quadratic, or cubic.

\*\* Allowance was made in this column for the fact that more cells employed nickel positive grids than silver.



TABLE NO. XVII

CONTRAST VALUES  
(with respect to capacity)

EFFECT	STAND PERIOD				
	2 hours	4 days	6 days	8 days	10 days
A <sub>L</sub>	4.5	6.1	6.8	3.7	2.6
AC	-18.1	-13.6	-7.8	-10.0	-10.1
AQ	-14.0	-16.7	-6.6	-10.7	-16.4
B <sub>L</sub>	6.5	2.0	-12.9	6.9	5.3
BQ	0.9	3.2	1.2	1.0	-1.3
C <sub>L</sub>	1.0	-1.7	0.2	0.7	-1.0
CQ	-1.1	-4.3	1.1	0.8	-4.3
D	-7.8	-6.8	-7.1	-2.3	-2.8
E <sub>L</sub>	-3.3	-6.4	-12.6	-8.8	-4.8
EQ	0.7	-3.7	3.1	0.5	-1.3
F <sub>L</sub>	-8.7	-1.4	-7.9	-8.7	-3.8
FQ	-0.8	-6.8	-2.1	-3.5	-5.0
G <sub>L</sub>	-5.2	-6.1	-1.1	-3.3	-0.6
GQ	4.7	5.2	-5.8	-0.3	-0.9
H	-5.1	-4.0	-8.7	-7.4	-2.5

TABLE NO. XVIII

CONTRAST VALUES  
(with respect to voltage)

EFFECT	STAND PERIOD				
	2 hours	4 days	6 days	8 days	10 days
A <sub>L</sub>	19.0	21.9	21.9	34.0	24.3
AC	-56.1	-46.1	-48.0	-45.2	-35.8
AQ	-21.2	-40.9	-38.9	-39.3	-41.9
B <sub>L</sub>	4.7	-12.4	-6.5	-14.2	-11.1
BQ	0.3	-1.1	16.6	1.3	4.8
C <sub>L</sub>	-2.3	19.8	3.2	44.3	-1.6
CQ	0.5	-3.9	0.6	2.1	13.0
D	9.1	17.5	24.6	11.0	8.8
E <sub>L</sub>	0.8	-27.5	2.2	-2.3	-4.6
EQ	0.4	2.27	2.7	9.6	2.5
F <sub>L</sub>	-24.6	7.2	-22.1	-19.1	1.2
FQ	1.1	0.1	-2.2	-0.9	-2.3
G <sub>L</sub>	0.5	-26.9	-2.9	-0.4	2.3
GQ	-5.9	-13.1	-7.1	-7.4	11.4
H	0.0	-1.9	-3.6	-2.2	-1.3

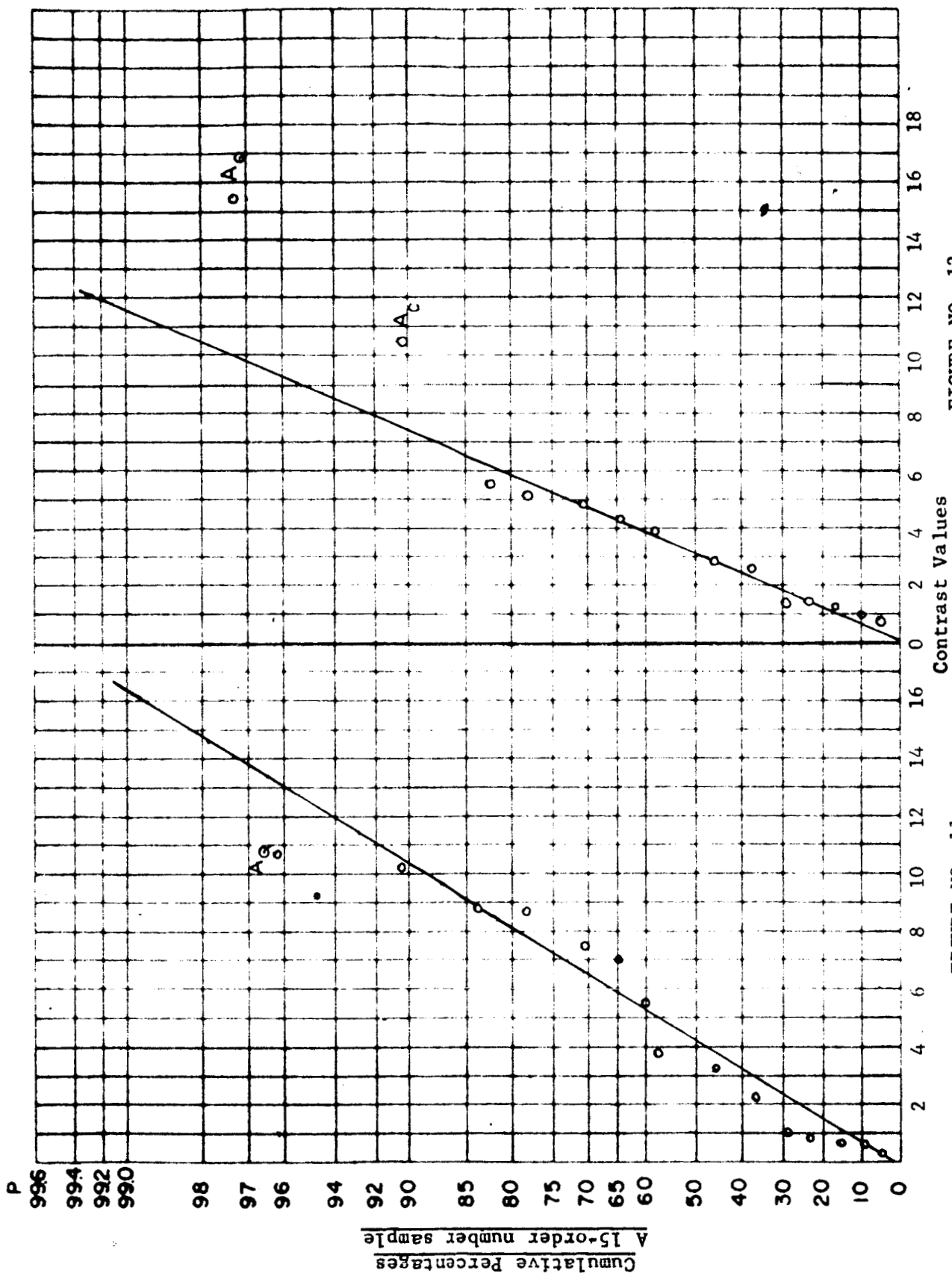


FIGURE NO. 11  
(8-day stand)

FIGURE NO. 12  
(10-day stand)

SIGNIFICANCE OF EFFECTS  
WITH RESPECT TO CAPACITY

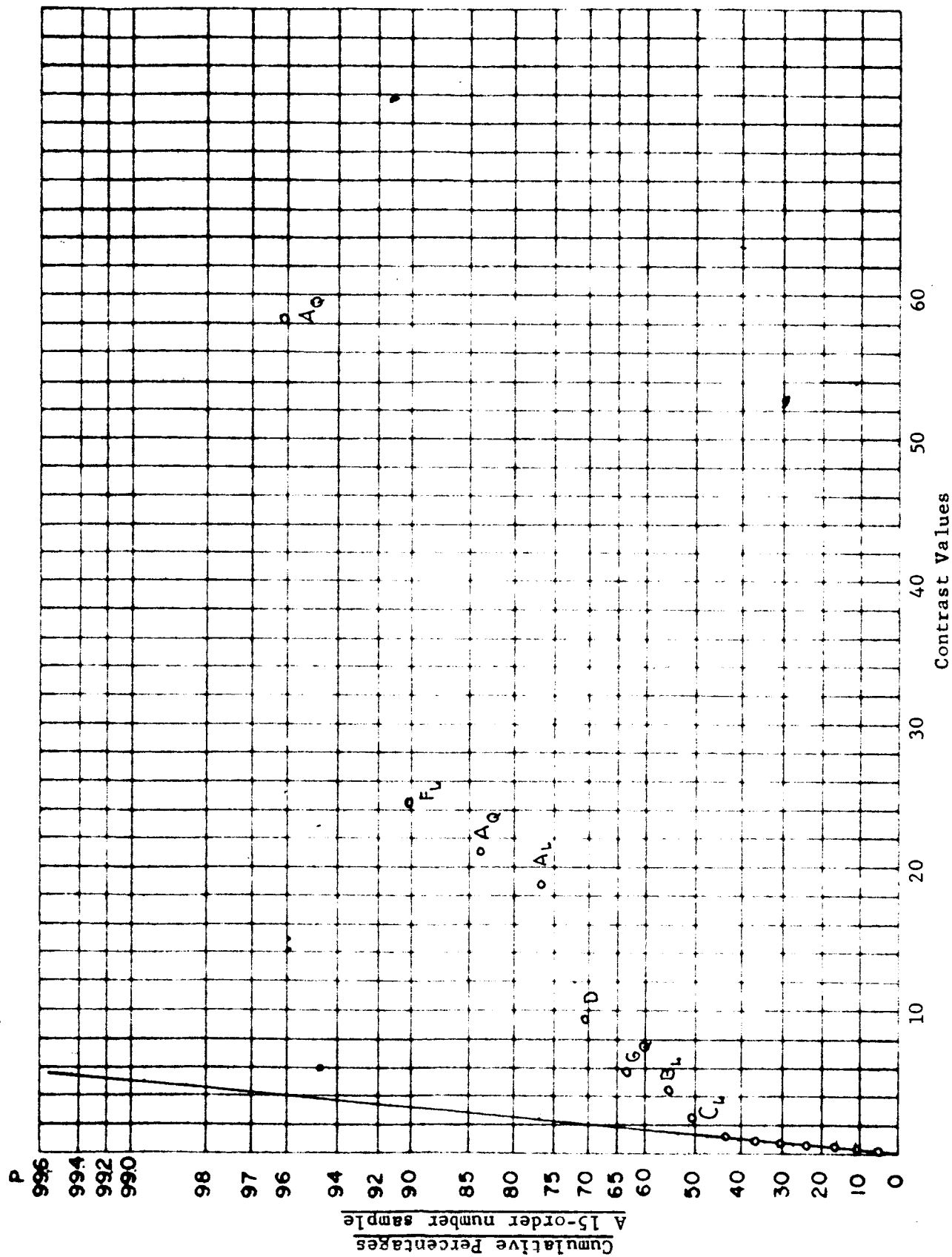


FIGURE NO. 13  
SIGNIFICANCE OF EFFECTS  
WITH RESPECT TO VOLTAGE  
2-HOUR STAND

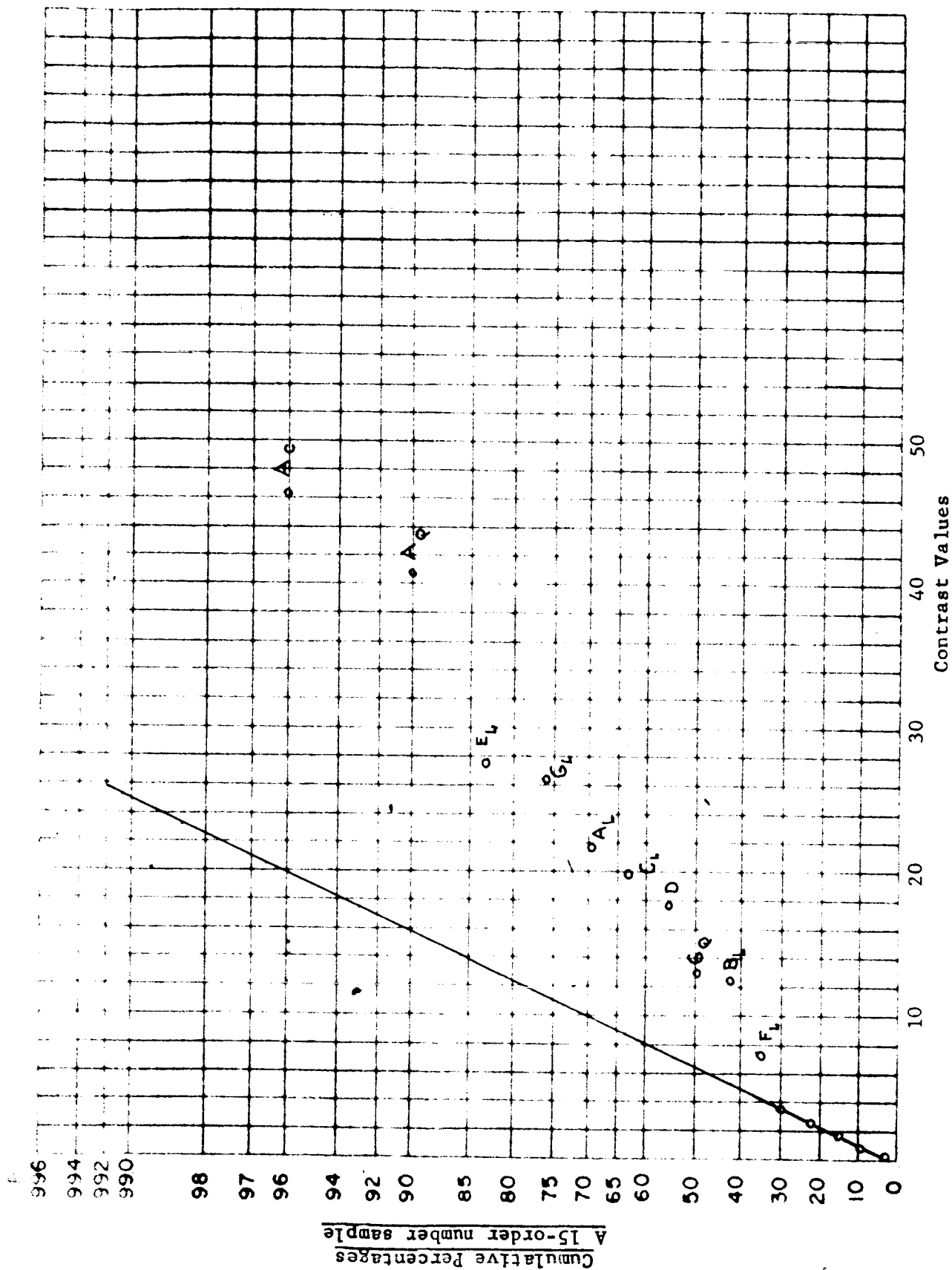


FIGURE NO. 14  
SIGNIFICANCE OF EFFECTS WITH RESPECT TO VOLTAGE  
4-DAY STAND

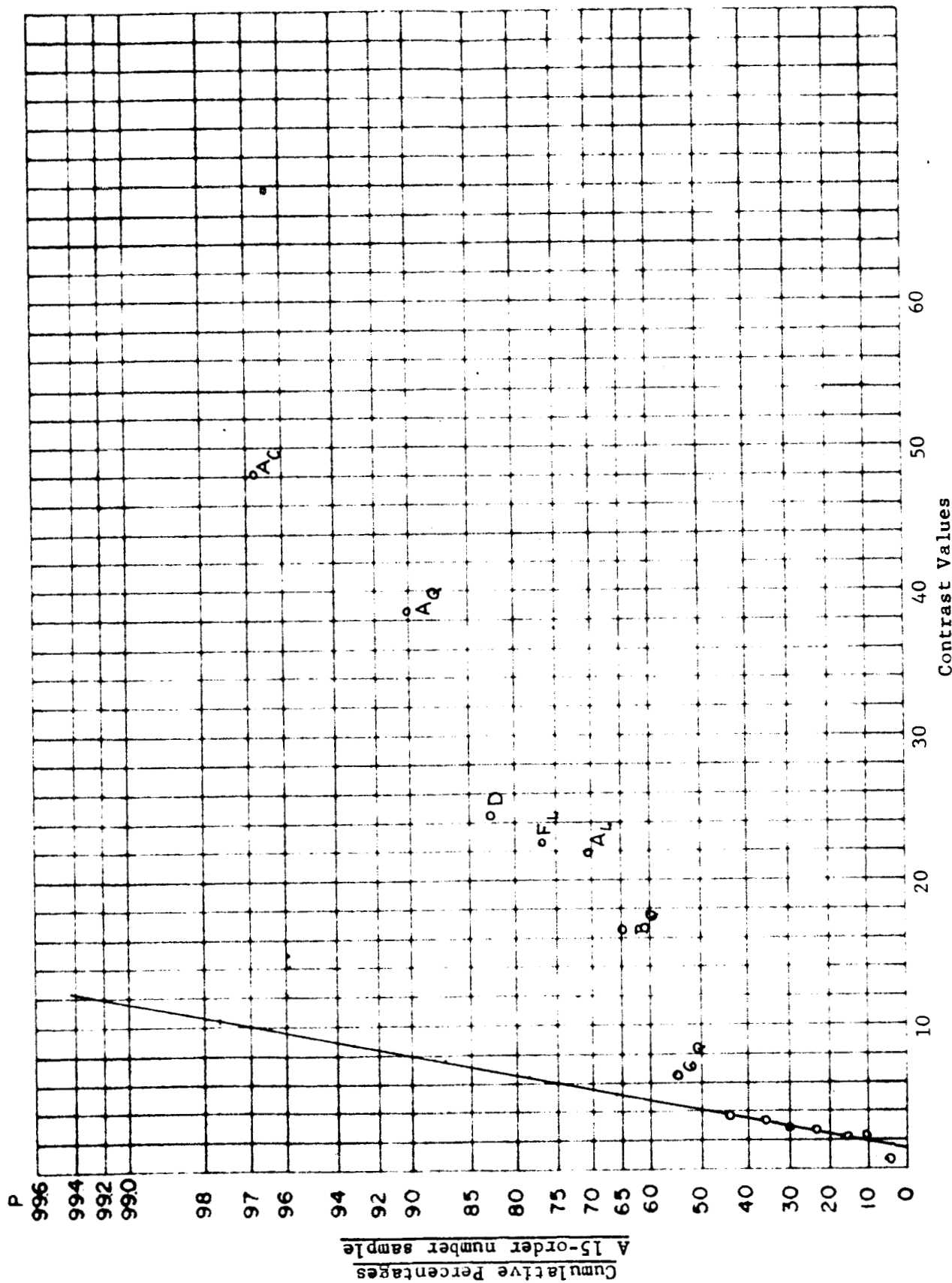


FIGURE NO. 15  
SIGNIFICANCE OF EFFECTS WITH RESPECT TO VOLTAGE  
6-DAY STAND

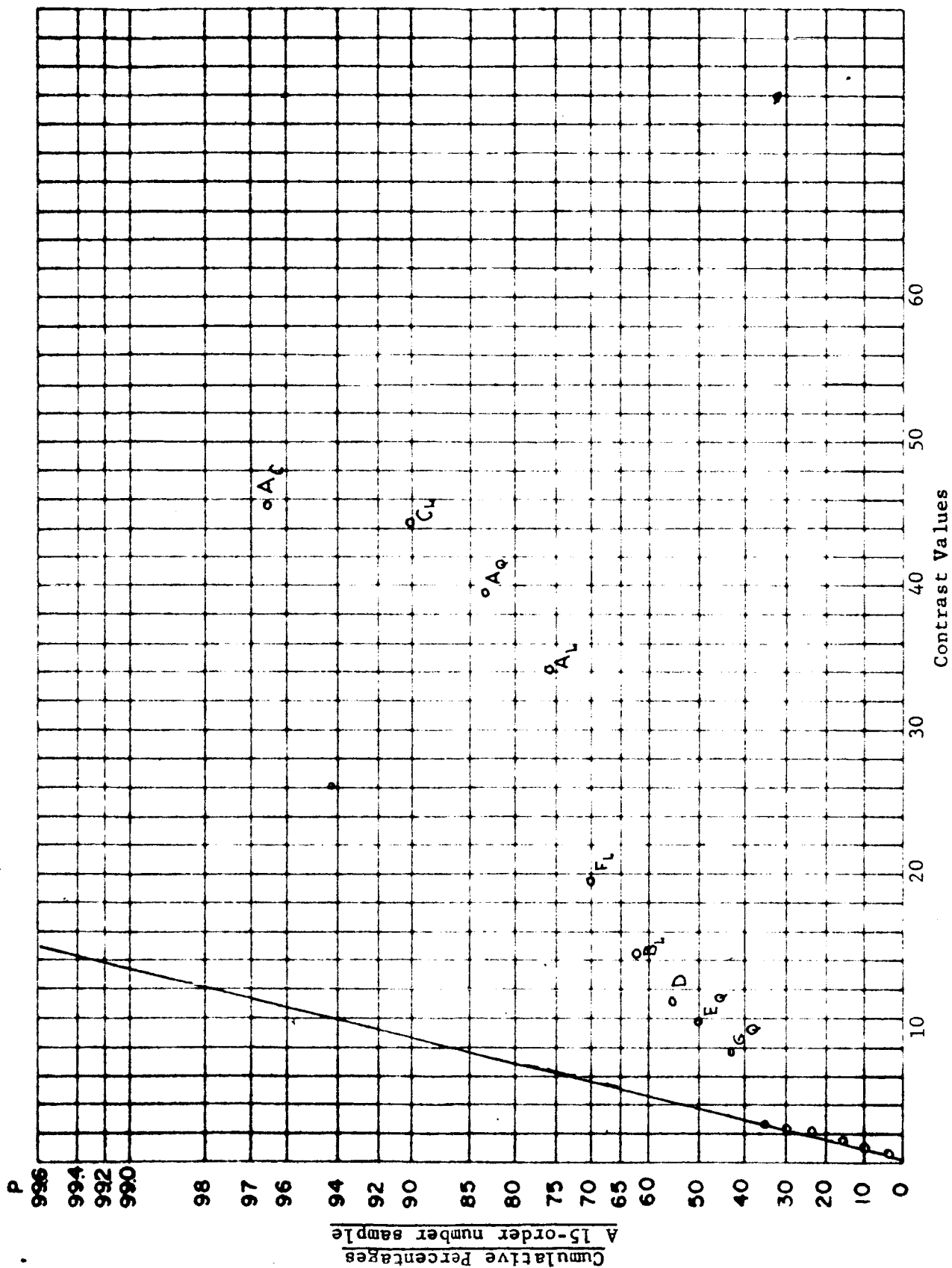


FIGURE NO. 16  
SIGNIFICANCE OF EFFECTS WITH RESPECT TO VOLTAGE  
8-DAY STAND

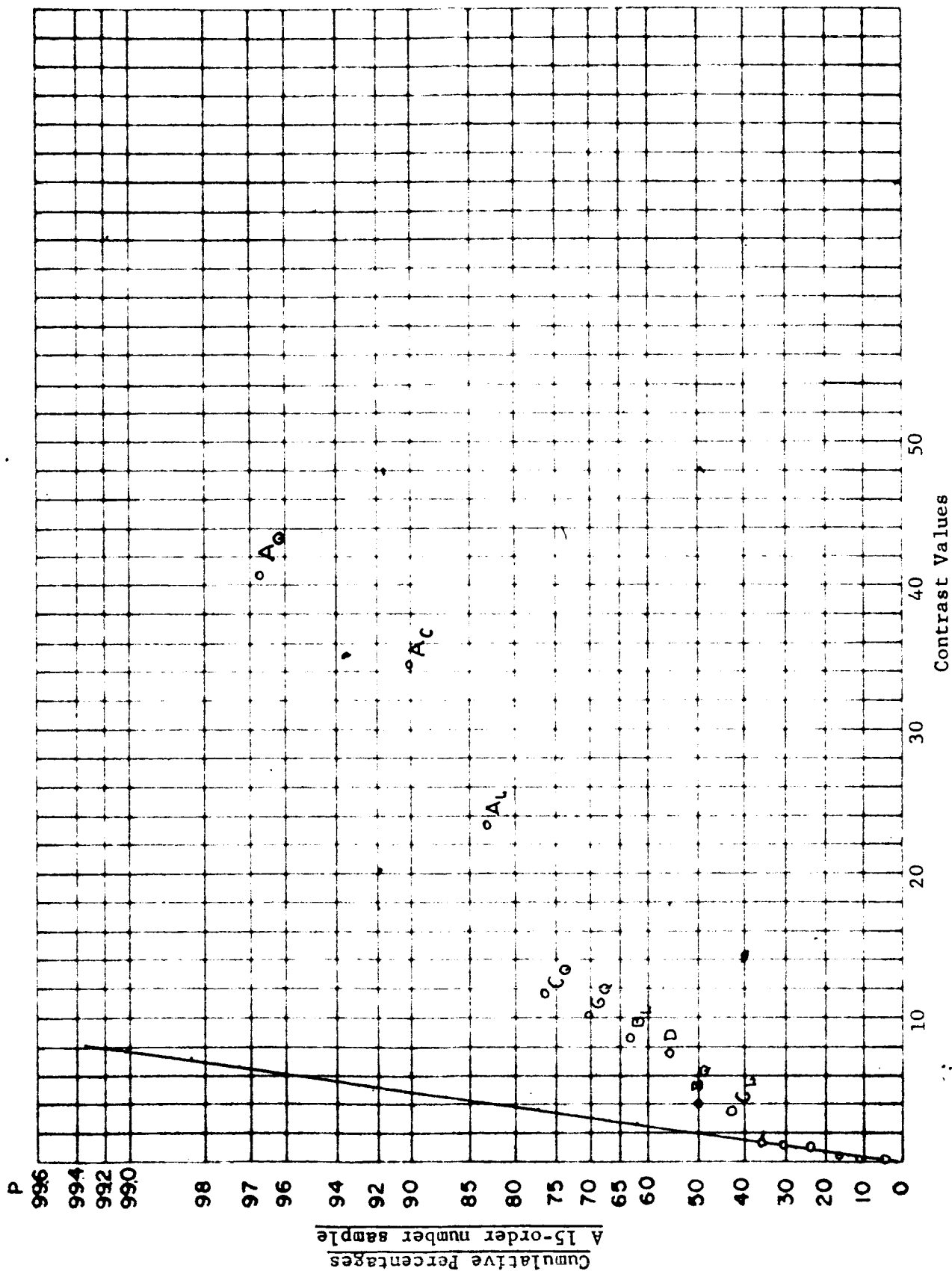


FIGURE NO. 17  
SIGNIFICANCE OF EFFECTS WITH RESPECT TO VOLTAGE  
10-DAY STAND

TABLE NO. XX

Values Used in Calculating Voltage Response  
By Use of Least Squares Equation (1)

TERM	2 Hr.	4 Days	6 Days	8 Days	10 Days
$\mu$	91.103	76.866	84.841	72.325	80.733
$a_0$	-0.916	-5.975	-0.284	-5.770	-4.333
$a_1$	1.758	3.325	-1.598	5.209	3.000
$a_2$	0.577	5.958	-0.760	3.997	1.060
$a_3$	-1.419	-3.308	2.639	-3.437	0.267
$B_1$	0.187	-7.083	-1.202	-6.231	3.816
$B_2$	-0.477	1.983	-0.416	0.499	-3.116
$\gamma_1$	2.340	5.550	4.243	11.183	8.433
$\gamma_2$	-0.929	0.649	-1.846	-2.008	-3.766
$d_0$	-2.069	-4.649	-2.221	-0.685	-2.483
$d_1$	2.069	4.649	2.221	0.685	2.483
$\delta_1$	-0.342	-2.350	5.327	-5.097	0.333
$\delta_2$	-0.262	1.283	-2.510	3.289	-0.800
$K_1$	3.869	18.499	-5.852	15.088	2.383
$K_2$	-1.360	-7.633	3.259	-5.463	-0.083
$g_0$	0.331	2.200	1.343	2.779	-1.179
$g_1$	-0.562	-0.233	-2.090	-3.163	1.089
$g_2$	0.232	-1.966	0.747	0.384	0.090
$h_1$	-0.005	-0.099	0.368	0.079	0.084
$h_2$	0.005	0.0099	-0.368	-0.079	-0.084



TABLE NO. XXI

RESPONSES (voltage\*\*)

Cell No.	2 Hr.	4 Da.	6 Da.	8 Da.	10 Da.
2	96.0	86.0	85.3	84.0	84.0
3	88.7	78.7	81.3	73.3	76.0
4	92.7	86.0	85.0*	84.0	84.7
6	94.7	89.3	86.0	80.7	82.7
7	96.0	92.0	88.0	85.3	88.7
8	90.7	79.3	82.0	78.7	83.3
10	96.0	92.0	92.0	88.0	89.3
12	90.7	76.0	76.0	74.0	79.3
13	90.7	84.7	85.3	82.0	86.0
14	94.7	95.3	92.0	89.3	89.3
17	87.3	77.3	82.0	73.3	82.0
18	93.3	85.3	85.3	74.7	77.3
19	90.7	88.0*	84.0	80.0*	77.3
20	91.3	84.0*	81.3	77.3	82.0
23	92.0	80.7	85.3	80.0	82.7
24	89.3	76.0	80.7	75.3	79.3
25	92.0	84.7	86.7	84.0*	82.0*
27	90.7	79.3	79.3	76.3	75.0*

\* Responses for these cells (which failed to deliver usable capacity) were estimated from the response pattern.

\*\*These responses are obtained by dividing the cell discharge voltage after 15 seconds by 1.50 volts. This artificial response facilitates handling of data.

FIGURE NO. 18

Predicted Capacity Response  
Following Eight Days Activated Stand at 130°r

Capacity (Minutes on Discharge at 30 Amps)

CELL NUMBERS

2 3 4 6 7 8 10 12 13 14 17 18 19 20 23 24 25 27

22  
20  
18  
16  
14  
12  
10  
8

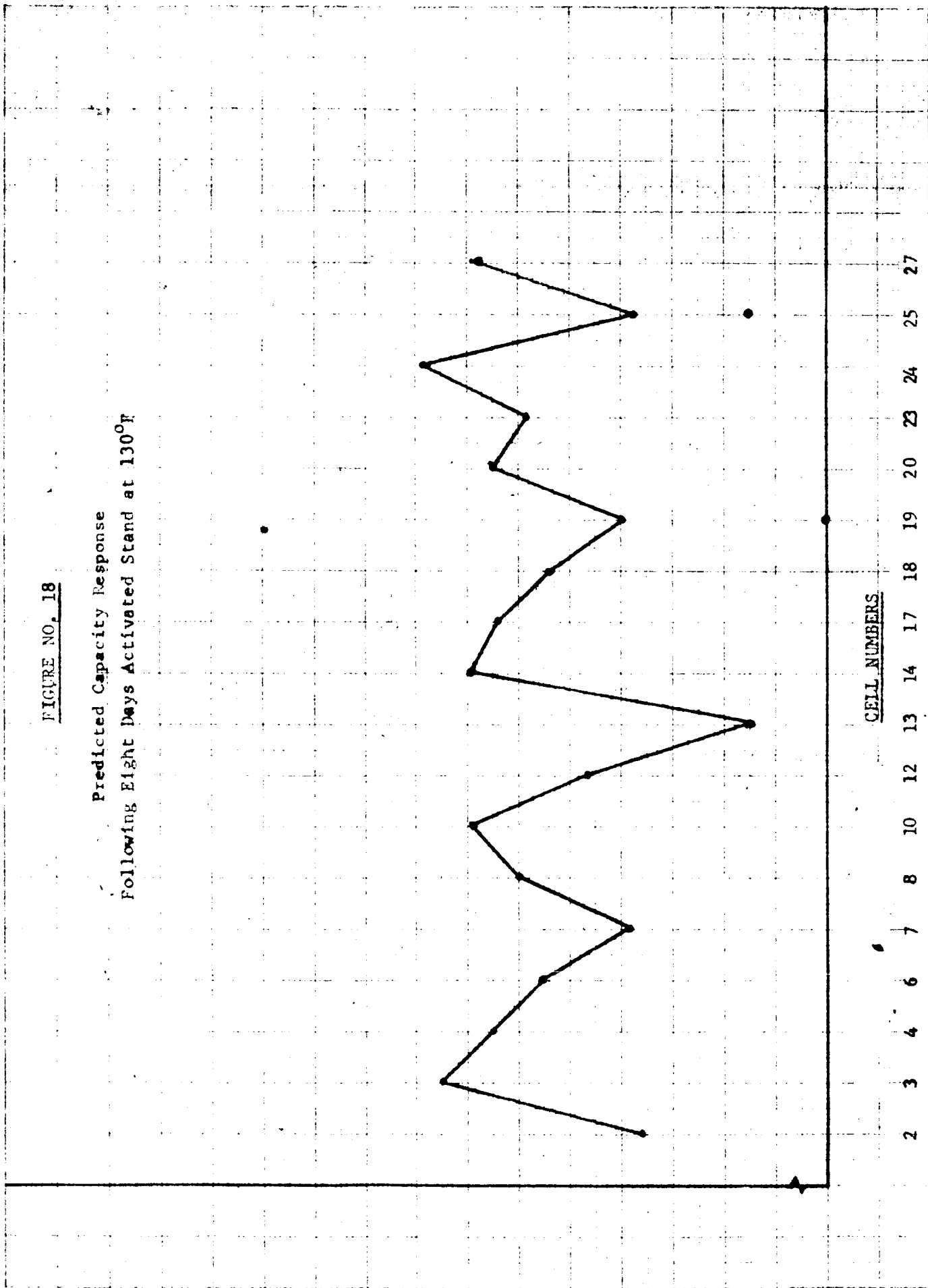


FIGURE NO. 19

Predicted Voltage Responses

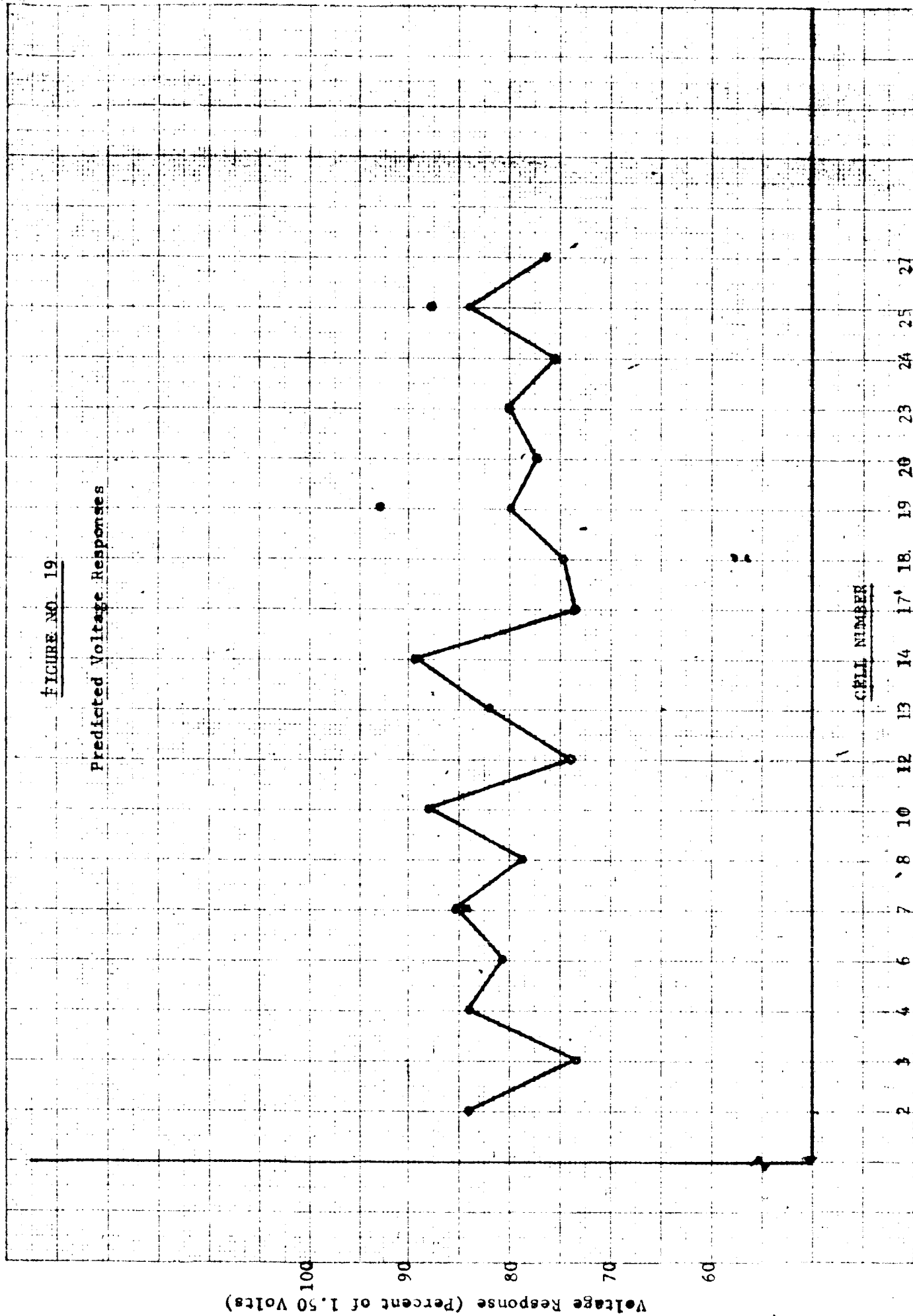


TABLE NO. XXII

OPEN-CIRCUIT VOLTAGES ON STAND  
 Preprototype Cell  
Silver Positive Grid

Cell No.	DAYS ON STAND							
	3	4	5	6	7	10	12	17
2	1.85							
3	1.85	1.84						
4	1.85	1.84	1.84					
5	1.85	1.84	1.84					
6	1.85	1.84	1.84	1.84	1.84	1.84	1.83	
7	1.84	1.84	1.84	1.84	1.84	1.84	1.84	
8	1.84	1.84	1.84	1.84	1.84	1.84	1.84	1.83
9	1.85	1.84	1.84	1.84	1.84	1.84	1.84	1.68
10	1.85	1.84	1.84	1.84	1.84	1.84	1.84	1.74
11	1.85	1.84	1.84	1.84	1.84	1.84	1.78	1.82
Mean	1.85	1.84	1.84	1.84	1.84	1.84	1.83	1.77

TABLE NO. XXIII

OPEN-CIRCUIT VOLTAGES ON STAND  
 Preprototype Cell  
Nickel Positive Grid

Cell No.	DAYS ON STAND							
	3	4	5	6	7	10	12	17
12	1.85							
13	1.85	1.85	1.84					
14	1.85	1.84	1.81	1.81	1.81			
15	1.84	1.82	1.80	1.80	1.80			
16	1.85	1.84	1.83	1.83	1.82	1.72	1.69	
17	1.84	1.84	1.81	1.81	1.81	1.74	1.65	
18	1.85	1.84	1.84	1.84	1.82	1.67	1.58	0.22
19	1.85	1.84	1.84	1.84	1.83	1.72	1.58	0.22
20	1.85	1.84	1.81	1.81	1.81	1.74	1.61	1.56
21	1.84	1.84	1.82	1.82	1.81	1.73	1.67	1.56
22	1.84	1.84	1.84	1.58	1.58	1.58	1.58	1.56
Mean	<1.85	1.84	1.82	>1.79	>1.78	1.70	1.62	1.03

FIGURE NO. 20

Discharge Characteristics Following Activated Stand at

Three Days at 130°

(Silver vs Nickel Positive Grid)

DISCHARGE VOLTAGE

1.6

1.5

1.4

1.3

1.2

1.1

1.0

Silver Positive Grid

Nickel Positive Grid

14

16

18

20

22

24

26

28

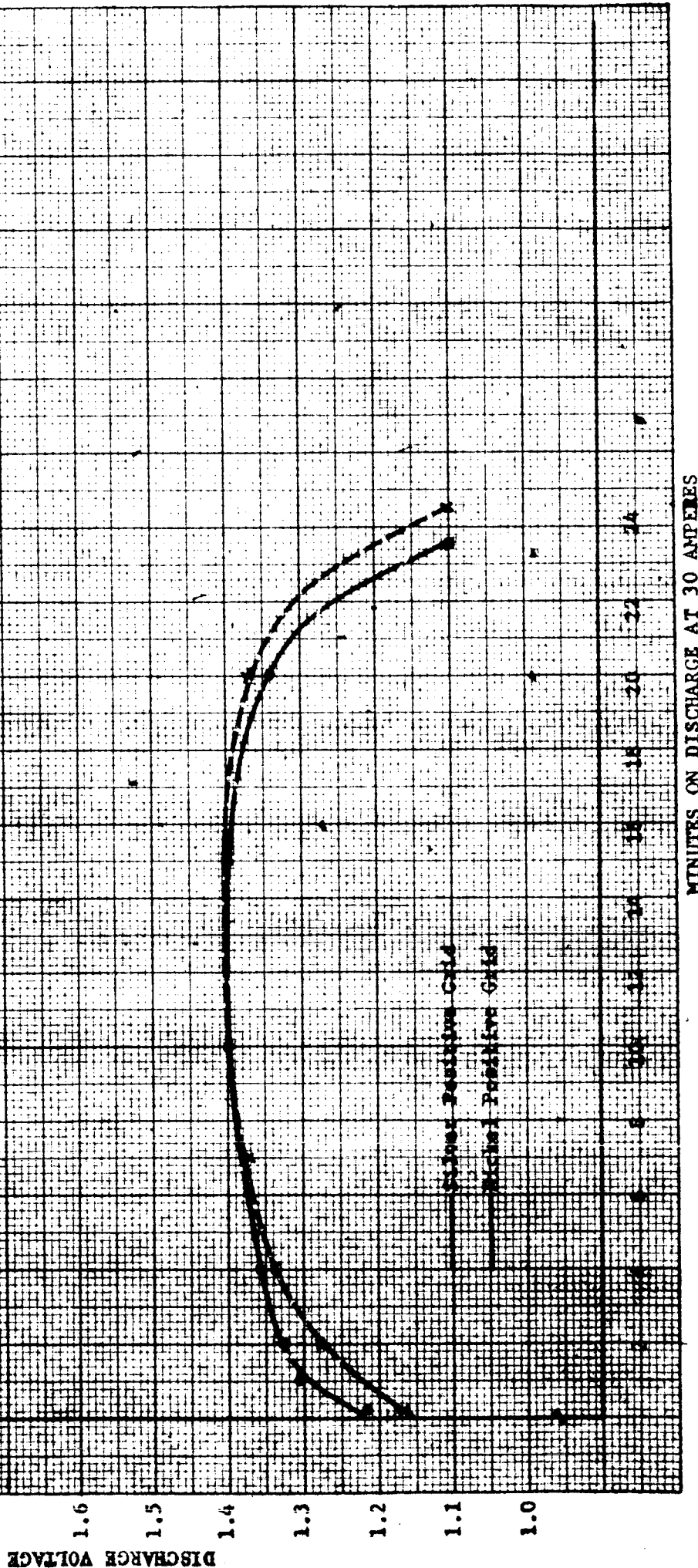
30

32

34

MINUTES ON DISCHARGE AT 30 AMPERES

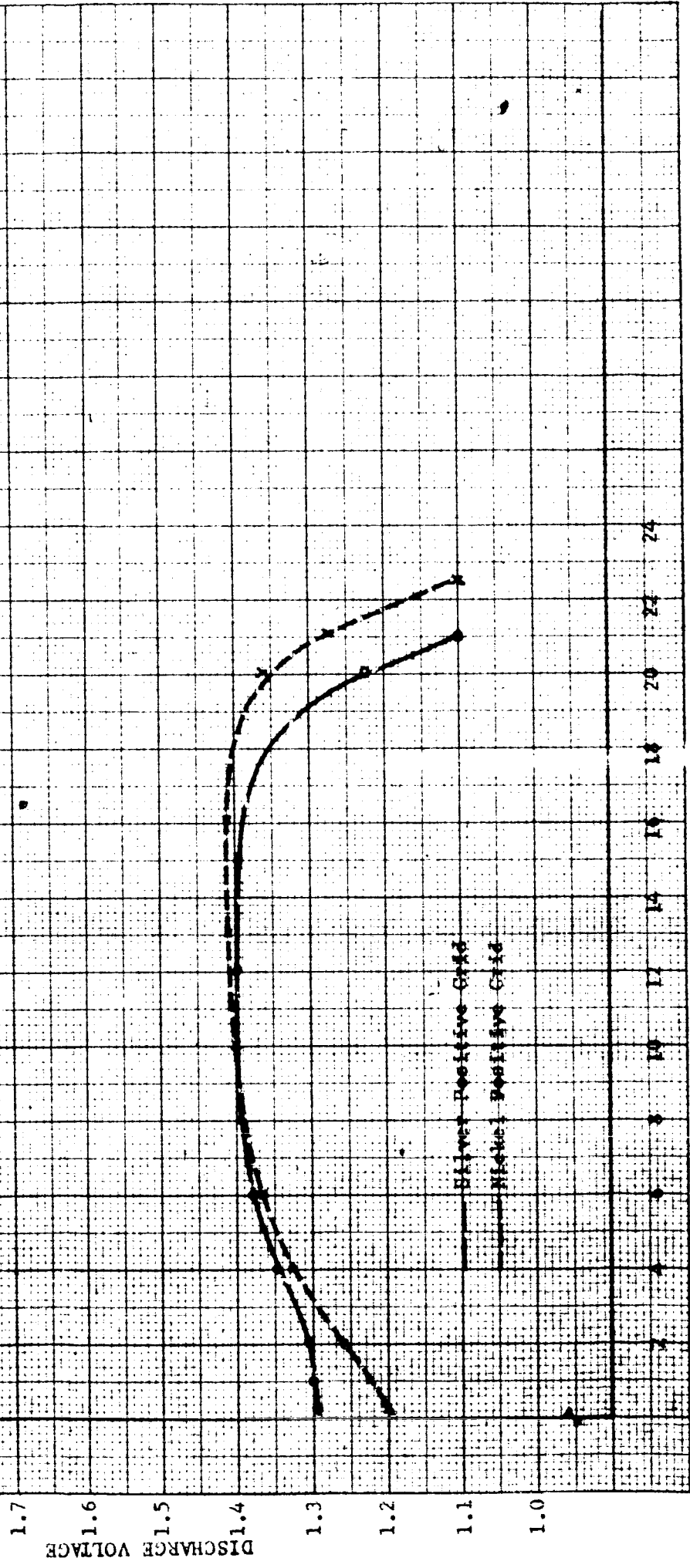
FIGURE NO. 21  
Discharge Characteristics following Activated Stand at  
FIVE DAYS 130° F  
(Silver vs Nickel Positive Grid)



MINUTES ON DISCHARGE AT 30 AMPERES

FIGURE NO. 22

Discharge Characteristics following Activated Stand at  
Seven Days at 130°F  
(Silver vs Nickel Positive Grid)



MINUTES ON DISCHARGE AT 30 AMPERES

FIGURE NO. 23

Discharge Characteristics Following Activated Stand at  
Eleven Days at 130° F  
(Silver vs Nickel Positive Grid)

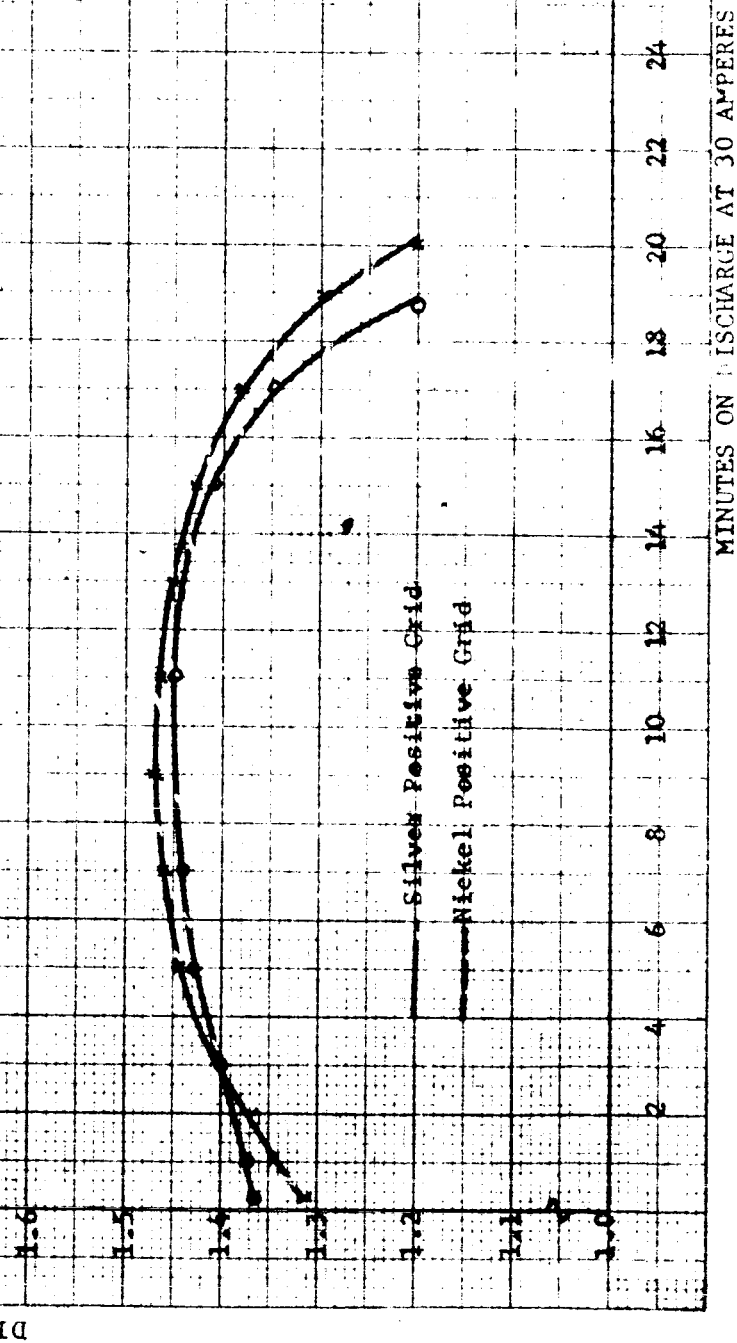
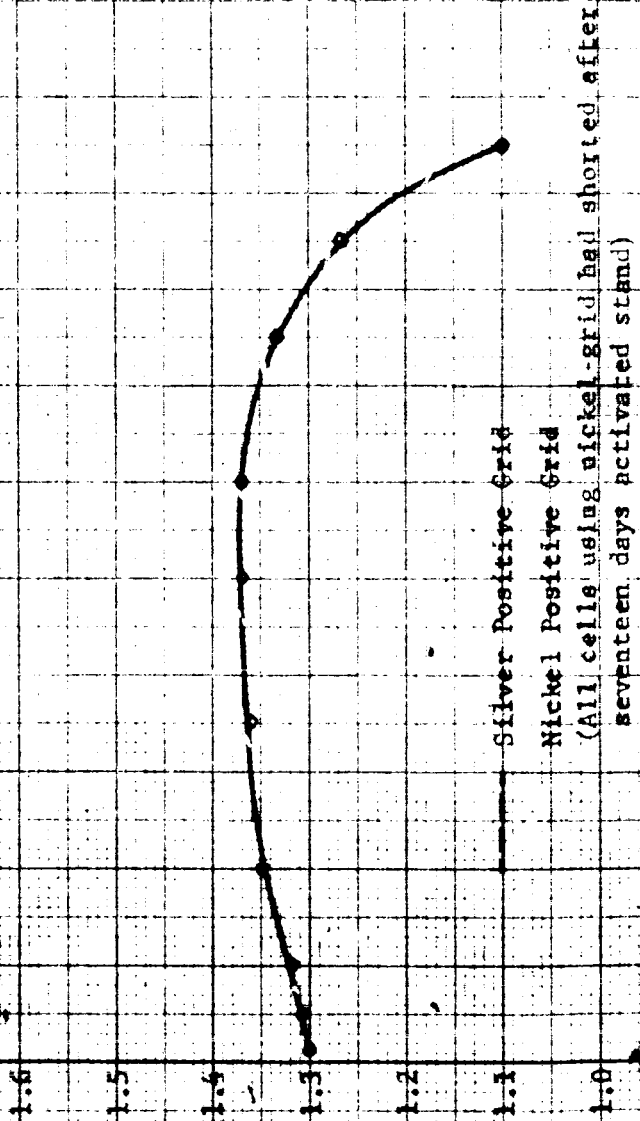




FIGURE NO. 24

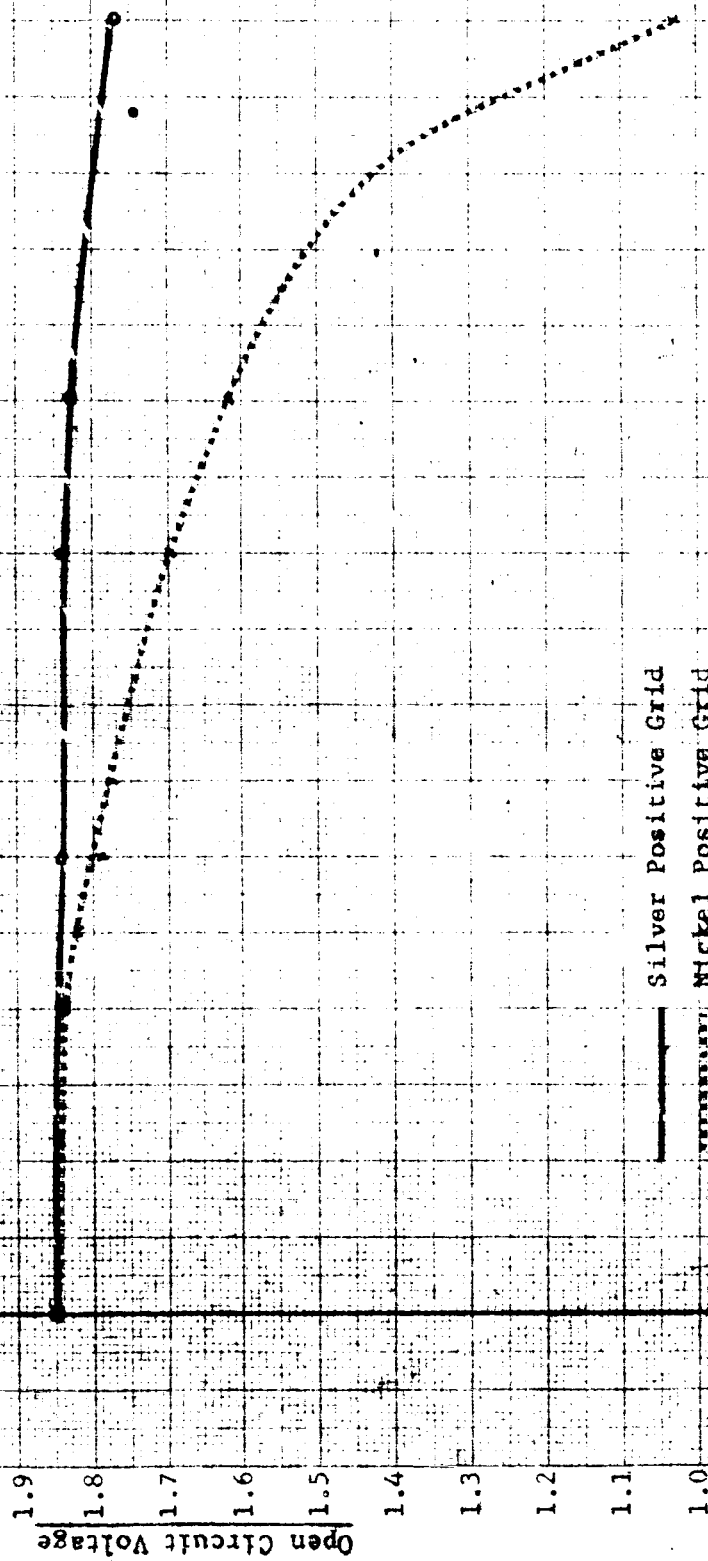
Discharge Characteristics Following Activated Stand at  
Seventeen Days at 110°  
(Silver vs Nickel Positive Grid)



MINUTES ON DISCHARGE AT 30 AMPERES

FIGURE NO. 25

Open-Circuit Voltage Retention at 130°F  
(Silver vs Nickel Positive Grid Metal)



DAYS ON STAND AT +130°F

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